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Official Publication of the Proceedings of the American Association of Textile Chemists and Colorists

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American Dyestuff Reporter was founded in 1917 and the Textile Colorist and Converter (formerly Textile Colorist) was incorporated with it in January, 1949. Published every other Monday by the **HOWES PUBLISHING COMPANY, INC., 44 E. 23rd Street, New York 10, N. Y. C R Howes,** President; **Ernest J Finan,** Executive Vice-President; **Norman A Johnson,** Vice-President & Treasurer; **Myron D Reeser,** Vice-President; **C Essenbreis,** Secretary

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May 12, 1952
VOLUME 41 NUMBER 10

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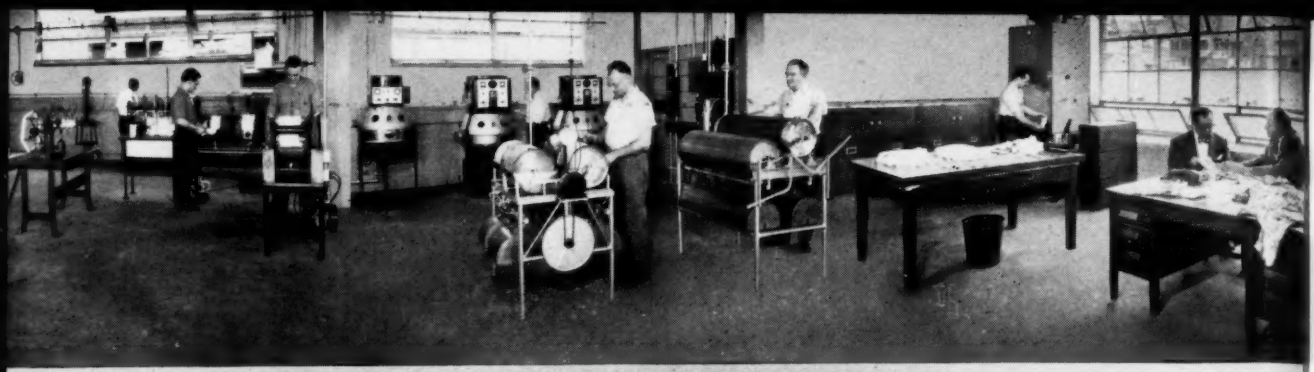

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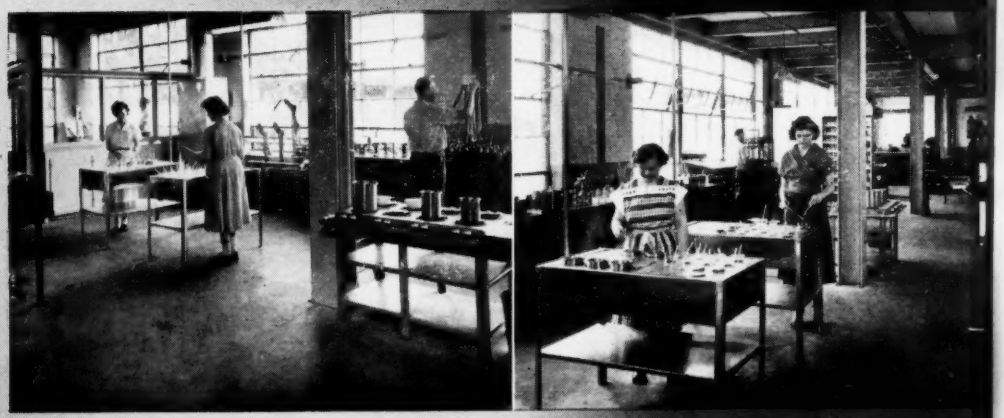

Proceedings of the
American Association of Textile Chemists and Colorists

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Editor of Proceedings: Carl Z Draves, 30 Lahey St, New Hyde Park, N Y

May 12, 1952

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CALENDAR

COUNCIL
June 13 (New York); September 19 (Philadelphia); November 6 (Boston)

GENERAL RESEARCH COMMITTEE
(See Council dates)

NATIONAL CONVENTIONS
November 6-8, 1952 (Hotel Statler, Boston); September 17-19, 1953 (Hotel Conrad Hilton, Chicago); 1954 (Atlanta, Ga).

HUDSON-MOHAWK SECTION
June 20 (Annual Outing).

MID-WEST SECTION
June 14 (Lake Lawn Lodge, Delavan, Wis); October 4 (Hotel Bismarck, Chicago).

NEW YORK SECTION
May 16 (Swiss Chalet, Rochelle Park, N J); June 13 (Outing, North Jersey Country Club, Wayne, N J).

NORTHERN NEW ENGLAND SECTION
May 16 (Andover Country Club, Andover, Mass); June 6 (Outing, Merrimack Valley Country Club, Methuen, Mass); December 6 (Annual Meeting, Lowell Textile Institute).

PHILADELPHIA SECTION
June 13 (Outing, Lu Lu Temple Country Club, North Hills, Pa).

PIEDMONT SECTION
June 6-7 (Ocean Forest Hotel, Myrtle Beach, S C); Sept 13 (Hotel Charlotte, Charlotte, N C).

RHODE ISLAND SECTION
May 23 (Providence Engineering Society); June 6 (Outing, Wannamoisett C C); Oct 24 (Providence Engineering Society); December 5 (Annual Meeting, Sheraton-Biltmore, Providence, R I).

SOUTH CENTRAL SECTION
May 17 (Patten Hotel, Chattanooga, Tenn); Aug 22-23 (Outing, Chattanooga Golf & Country Club); December 6 (Patten Hotel).

SOUTHEASTERN SECTION
June 6-7 (Outing, Radium Springs, Ga); Sept 13 (Biltmore Hotel, Atlanta).

WESTERN NEW ENGLAND SECTION
June 20 (Outing); Oct 3, Nov 14 and Dec 12 (Rapp's Restaurant, Shelton, Conn).

OTHER EVENTS

ACHEMA X
Exhibition Meeting for Chemical Engineering, May 18-25, Frankfort/M, Germany.

AMERICAN CHEMICAL SOCIETY
7th National Chemical Exposition, Sept 9-13, Chicago Coliseum, Chicago, Ill. This will include the 2nd International Art Exhibit.

AMERICAN OIL CHEMISTS SOCIETY
Short Course industrial trips, July 8-9, 10-11.

AMERICAN SOCIETY FOR QUALITY CONTROL
Sixth Annual Convention, May 22-24, Syracuse, New York.

AMERICAN SOCIETY FOR TESTING MATERIALS
Annual Meeting, June 23-27, 1952, New York, N Y. (The biennial Apparatus and Photographic Exhibits will be held in conjunction with this meeting).

AMERICAN STANDARDS ASSOCIATION
3rd National Standardization Conference, Sept 8-10, Museum of Science and Industry, Chicago, Ill.
34th Annual Meeting, November 19, Waldorf-Astoria, New York, N Y.

AMERICAN TEXTILE MACHINERY ASSOCIATION
Exhibit of American Textile Machinery, April 26-May 1, 1954, Atlantic City Exhibition Hall, Atlantic City, N J.

CANADIAN TEXTILE SEMINAR
September 11-13, Queen's University, Kingston, Ont.

FIBER SOCIETY
Sept 10-11 (Princeton, N J).

INTERNATIONAL ORGANIZATION for STANDARDIZATION
Meeting of Technical Committee 38 (Textiles). Subcommittees 2 (Shrinkage of Fabrics in Washing) and 6 (Fiber Testing)—June 13, 14 and 16, Columbia University, New York, N Y.

LEIPZIG FAIR
September 7-17, Leipzig, Germany.

NATIONAL ASSOCIATION OF HOSIERY MANUFACTURERS
41st Knitting Arts Exhibition, April 27-May 1, 1953, Atlantic City Auditorium, Atlantic City, N J.

NEW YORK BOARD OF TRADE
26th Annual Meeting: September 25-28, Pocono Manor Inn, Pocono Manor, Pa.

SOCIETE DE CHEMIE INDUSTRIELLE
25th International Congress for Industrial Chemistry, May 18-25, Paris, France.

TEXTILE INSTITUTE
Annual Conference, June 2-6, Edinburgh, Scotland.

EMPLOYMENT REGISTER

This column is open for two insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the Secretary of the Association, Lowell Textile Institute, Lowell, Mass. It is understood that these will be open to inspection by prospective employers who can obtain further information from the Secretary.

52-14

Education: B S Textile Chemistry & dyeing; M S Organic Chemistry.
Experience: Textile research chemist.
Age: 27; single; references; New England preferred but not essential.

4-28, 5-12

PRELIMINARY INVITATION TO CONTRIBUTE TECHNICAL PAPERS AT 1952 CONVENTION

PREPARATIONS for the technical program at the 1952 National Convention, to be held in Boston, Mass, Nov 6-8, are now underway. Persons desiring to present a paper should proceed according to the following established instructions and regulations:

(1) Notify Ernest R Kaswell, Convention Technical Chairman, Fabric Research Laboratories, Inc, 665 Boylston St, Boston 16, Massachusetts, as soon as possible, giving the title of the proposed paper. The time required for the presentation should be stated (30-45 minutes).

(2) When the number of papers available is known, a selection will be made by the Committee, and the contributors will be advised as to whether their papers have been accepted. The program must be completed as soon as possible in order to assure the best possible arrangement of the papers and to provide adequate time for publicity.

(3) An abstract of the paper will be required in advance of the program. Contributors will be notified of the date on which this abstract is required.

(4) Copies of the completed paper must be in the hands of the Technical Program Chairman by the first week of September so that the necessary arrangements for prompt publication may be set in motion. Earlier submission will be appreciated.

(5) Papers must not be primarily of advertising or promotional character. However, the subject matter may consider commercial products, machinery, instruments, or processing procedures of a commercial nature, provided that they are evaluated from a technical viewpoint.

(6) The Technical Program Committee must be the final judge as to which papers are to be accepted, but every effort will be made to include all suitable contributions in the program.

PUBLICATION OF COLOUR INDEX TENTATIVELY SET FOR 1955

ACCORDING to the Annual Report of the Council of the Society of Dyers and Colourists presented at the Annual General Meeting of the Society held on March 28th the tentative date for publication of the new *Colour Index* has been set for 1955. That section of the report which deals with the *Colour Index* is reproduced herewith.

"COLOUR INDEX (SECOND EDITION)—There have been no further changes in the personnel of the Editorial Panel, and members have had a very busy and eventful year. The normal editorial work has involved the handling of a very large amount of information from cooperating dyemakers. It had originally been planned that 30th June 1951 should be the closing date for receipt of information from cooperating dyemakers, but it was necessary to extend this to 31st December, and the bulk of the material has now been received. Dye manufacturers continue to take the keenest interest in the work, and they are doing everything possible to complete their contributions so as to ensure inclusion in the published work.

"This year was noteworthy in that we were privileged to have with us for discussion on the *Colour Index* Mr C Norris Rabold, President of the AATCC, Mr L S Little, Chairman of the AATCC General Research Committee, and Mr W D Appel of the National Bureau of Standards, Washington, a past president of the AATCC. Our joint discussions dealt with the cost of production, means of financing the work, and matters relating to editorial procedure. During the last few years book production costs have risen considerably owing to large increases in the price of paper and increased wages, and the effect of these increases has been that estimated production costs have risen by approximately 50%. Approximate estimates indicate that the gross production cost will be of the order of £85,000, and it was agreed with our American colleagues that a ceiling cost of £90,000 should be set, and that if necessary economies would be effected to keep within this sum.

"It is now possible to outline the form the new *Colour Index* will take. Part I will consist of three volumes, each of about 1000 pages, and will deal with approximately 5000 dye entities. The commercial names of each dye entity will be listed, with data on application, usage, and fastness properties. Part II will be a single volume of approximately 700 pages and will list the constitution of 3500-4000 dyes. Part III will be a single volume of approximately 400 pages, containing a general index to Parts I and II, and is expected to list the 35,000 commercial names of dyes and also an index for intermediates.

"The drawing up of the Agreement between the Association and the Society has been completed, and the formal signing took place on 18th January 1952. In agreement with our American colleagues a tentative production schedule has been drawn up on the basis that all data from dye manufacturers will have been received by the end of 1951. It is expected that compilation of these data will be well advanced during 1952 and printing will occupy a further two years. It will be seen, therefore, that the tentative date for publication is 1955.

"The present year will call for great efforts by the members of the Editorial Panel, and Council records its indebtedness to them for their past efforts and extends its good wishes for their future work. Council also wishes to thank those firms and institutions who have permitted and encouraged members of the Panel to take part in this work."

—♦ ♦—

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(Concluded on Page P304)

New York, Piedmont and Rhode Island Sections—

THE MARHEN PROCESS*

DONALD E MARNON
General Dyestuff Corporation

D E Marnon

In the application of vat dyes to textiles, an important consideration is proper and adequate reduction of the dyestuff without overreduction, without waste of chemicals and with maintenance of the solution in a condition favorable for good dyeing. Although authorities (*1*), such as Clark, Conant and others, have suggested the theoretical importance of a knowledge of oxidation-reduction (redox) potentials for an understanding of the reduction of quinones (many vat dyes are quinones), and Albert Schaeffer (*2*) has recently suggested the usefulness of the redox value for each vat dye in making formulations for vat dyeing, no one has yet made available to the dyer a simple and practical means whereby large-scale, and especially continuous, vat dyeing can be controlled with certainty of results. This control the author has now made available by his "Marhen Process."

The reducing condition of a vat dyebath is dependent upon three variables: namely, temperature, active alkalinity, or pH, and concentration of sodium hydrosulfite (*2*). But to derive good recipes or to control any process well with three variables is a difficult problem. By use of the Marhen Process, however, which gives continuously one potential reading, control is greatly simplified since the reducing condition of the bath is accurately reflected by only one value dependent upon the three variables.

The Marhen Process is based on the tendency of a reducing solution, such as a bath of hydrosulfite and caustic soda, to give up electrons as illustrated in the equation of the author. When a bright platinum electrode is placed in the bath, the electrons tend to escape from the solution at the electrode with a certain electrical pressure, or voltage. To measure this tendency electrically, a double connection is always necessary, and therefore a connection is made to the solution both through the platinum electrode and through a saturated calomel/potassium-chloride electrode, which has a fixed and known electrical potential at a given temperature. The Marhen apparatus measures, in a reliable and reproducible way, the voltage between these two electrodes, which then differs from the true redox potential of the bath by a known amount. For practical purposes the author uses the direct readings from his instrument without calculation. The Marhen process has succeeded in measuring these potentials practically by the application of a high-input impedance amplifier in such a way as to protect the platimum electrode from polarization and thus to obtain reliable and constantly recordable readings under commercial dyeing conditions.

In this first report written by the inventor himself about the Marhen Process, the practical consequences of its application to large-scale vat dyeing is discussed.

INTRODUCTION

THE MOST important factor in the application of vat dyes is that of reduction. Upon this single factor rests the success or failure of a particular operation.

What is reduction? How is this important factor defined? A standard dictionary defines reduction as "The act or process of reducing in any sense". It then lists many specific definitions according to the field of endeavor. For example, medical reduction may refer to the setting of a fracture or the elimination of some abnormality. Mathematical reduction refers to changing the terms of a problem to equivalent terms more easily understood.

Chemistry also has its own special definition of reduction. Chemical reduction is defined as the addition of one or more electrons to the atom or molecule being reduced. You may ask, "How could an addition bring about a reduction?" This does sound ambiguous if one considers reduction in the common sense. In searching for a simple analogy that would clarify this concept, the thought of bills or debts came to mind. What one thing do we receive which does not add to our total resources, but actually causes them to diminish? Yes, bills. Thus, we may consider bills as negative charges which tend to reduce our over-all positive value. Returning to chemistry then, the electrons are the bills, which reduce the positive status of the atom or molecule. This is chemical reduction.

The application of vat colors, as we well know, involves the conversion of these colors to their soluble leuco form. This conversion is brought about by treating the dye with an alkaline solution of sodium hydrosulfite. A consideration of this reaction shows that it is an oxidation-reduction reaction, that is, the dye is reduced and the hydrosulfite is oxidized. This means that a transfer of electrons from hydro to dye has occurred. This reaction may be illustrated as follows, where "Ph" stands for the dye nucleus and "O=Ph=O" for the vat dye, which is often a quinone:

$$\text{I}\quad 4\overline{O}H + \overline{\overline{S_2O_4}} \rightarrow 2\overline{SO_3} + 2H_2O + 2e$$

$$\text{II}\quad O{=}Ph{=}O + 2e \rightarrow \overline{O}{-}Ph{-}\overline{O}$$

$$\text{III}\quad 4NaOH + Na_2S_2O_4 + O{=}Ph{=}O \rightarrow NaOPhONa + 2Na_2SO_3 + 2H_2O$$

Referring to this chart, we see three equations illustrated. The first two are ionic and consider only the essential groups involved in the reaction, while the third is the complete summation of the first two.

The first equation shows the reaction between caustic soda and hydrosulfite. The only important product of this reaction is the electron. It is the electron in equation II which reacts with the carbonyl group common to every vat dye and reduces it as we see on the right. The third equation shows the over-all effect. Here the sodium ions have been included, not that they contribute to the reduction, but we now have chemicals with which we are familiar instead of ions. We realize that this third equation represents a simplification of the actual fact. It is known that many other by-products do result from both the oxidation and decomposition of hydrosulfite; however, these by-products do not change the fundamental reaction of reduction.

The chemist would normally consider these equations, calculate the amounts of caustic and hydro required on a theoretical basis, attempt to determine the amount of dye in a commercial material and then

* Presented before the New York Section at Kohler's Swiss Chalet, Rochelle Park, N J on Nov 30, 1951, before the Piedmont Section at the Robert E Lee Hotel, Winston-Salem, N C, on Feb 2, and before the Rhode Island Section at Johnson-Hummock's Grille, Providence, R I, on Feb 29. Also presented by J Rath before the VTCC in Germany on May 3, 1952.

proceed to dye a sample of cloth. He might use an excess of hydro because he knows how unstable this material is. As many of you realize, the chances for success of such an operation are slim. Unless the chemist has considered the purity of hydro, caustic and dye, considered the stability of the caustic-hydro system at the temperature of dyeing, considered the amount of aeration dependent on machinery used and fabric construction, considered the amount of caustic consumed by the cloth, these and other items too numerous to mention but nevertheless important, he will undoubtedly have an inferior dyeing. Because it is almost impossible for the chemist or the dyer to know all of these variables, it has been necessary to establish formulas for practical application that are based on previous experience and know-how.

These dyeing formulas have been handed down from dyer to dyer through the years. Research and development by both the wet-processing concerns and the dyestuff manufacturers have modified them to meet the economies of high-speed production. In endeavoring to produce higher-quality materials, the dyer has used any means at hand to determine the condition and constancy of his dyebath. Both titration and indicator papers were introduced. The dyer is able to determine caustic in the bath by titration with acid. The amount of hydro is determined by titration with either iodine or indigo. Unfortunately, these values are a measure of unreacted chemicals. An approach to the measurement of the ability of the bath to reduce was the introduction of indicator papers. Vat Yellow G paper immersed in a dyebath turns blue if the bath is reductive. Consider, however, that this color change only indicates that Vat Yellow G, one of the most easily reduced vat dyes we know, will be reduced. It is quite possible that the dyestuff on the cloth will remain unreduced. What actually is needed is a means of directly measuring the reducing ability of the bath. We therefore need a direct measurement of the reaction products of caustic and hydro. Consider again equation I. On the right side we find sulfite, water and electrons. Determination of sulfite, while chemically possible, would be lengthy and unreliable. The measurement of water in a water system is of course ridiculous. This leaves the electrons. As they are directly responsible for reduction, what better choice for measuring the reduction potential of a dyebath?

EXPERIMENTAL

How do we measure this reduction potential? The following discussion is not intended as the absolute answer to this question. Our theories are based on the work that has been done to date. Future research may show that we have erred, but without a working hypothesis we have nothing.

MEASURING REDUCING POWER ——The electrons generated by the reaction of caustic and hydro are the quantities that we wish to measure. In that measurement we do not want to consume them but only to measure their tendency to react. As with other applications, such a measurement indicates a set of electrodes immersed in the bath, which are connected in turn to a meter. The selection of electrodes is governed only by their suitability in the chemical bath to be measured. The indicating electrode was selected of platinum because of the relative inertness of this material. For a reference electrode we selected the calomel rather than a hydrogen electrode because of its stability in caustic and hydro. Now we must connect to these electrodes some sort of an indicating device, bearing in mind that we do not wish to consume the electrons. We must avoid this consumption for two reasons: if they are consumed, first, we will get an erroneously low reading and, second, related electronic measurements tell us that electrode polarization and consequent loss of sensitivity will occur. Electronics also tell us that the only way to prevent this consumption is to put sufficient barriers or resistance in the way so that current flow or electron movement is practically impossible. Equipment that utilizes this principle is described as having a high-input impedance. Thus, we must connect our electrodes to a high-input-impedance indicator. The circuits involved in such an indicator I shall leave to someone much better qualified than I to discuss. The particular instrument which we first used employs the very high input-impedance amplifier of a Beckman pH meter connected in turn to a Brown ElectroniK recorder for recording the data. In Figure 1 is shown a Foxboro instrument now available for automatically recording redox potentials for the Marhen Process.

Thus far we have described what we believe is the essential mechanism of the reduction of vat dyes with caustic and hydro. We have also described an instrument which will measure the mechanism. Such an instrument placed in the hands of the dyer enables him to measure and control the chemical balances involved in dyeing with vat colors.

PRACTICAL APPLICATION

The rest of this paper will deal with the practical application of this process to the various methods that are used today in the application of vat colors to textiles. At the present time, about a half million yards has been dyed under the controlled conditions of this process. This yardage represents both continuous- and batch-dyeing methods. This portion of this paper will cover those trials conducted on continuous-dyeing ranges.

We have controlled three types of continuous dyeing: Williams unit/water-phase dyeing, pad-steam dyeing, and hot-oil dyeing. These will be taken up in the order mentioned.

WILLIAMS UNIT/WATER PHASE DYEING——Most of you are familiar with this system of continuous dyeing in which the key unit is the chemical reduction bath. By operating this bath at temperatures ranging from 160-200°F, one is able to dye continuously at rates up to 150 yards a minute. Proper control of the chemical balances in this unit are very important for several reasons: first, control is important to insure the proper development of the dyestuff. We shall show by examples that as much as 10% increase in color value has been obtained by establishing the proper balance and quantities of caustic and hydro. This means more yards per pound of dyestuff. Second, control is important to avoid wasting either caustic or hydro in maintaining the bath. Third, control is important to protect some dyes from damage due to overreduction. This problem presents quite a dilemma to the dyer. Early in the developmnt of the Williams Unit it was found that material developed in a unit which contained only caustic and hydro was lighter in depth at the beginning than later in the run. This is known as tailing. Obviously, color was stripped from the initial yards and dyed on later yards until a dye equilibrium was established in the bath. This situation was eased considerably by adding color to the unit prior to running. The amount added was based on prior experiences and represented an attempt to establish equilibrium conditions from the very beginning. Now we have color reduced in the unit and color being carried in by the cloth to be reduced. In many cases the dyestuff used is sufficiently stable in its reduced form to exist for long periods without damage. There are many dyes, however, whose leuco forms are quite unstable at these high temperatures. Here then is the dilemma. How can we efficiently reduce the dye that is carried in by the fiber without overreducing dye that remains in the bath?

The Marhen Process stemmed from the last of these three reasons and was gradually expanded to cover the other two. In the search for some way to dye the colors, notably Vat Blue BC, that are sensitive to high temperature in their leuco

form, our Charlotte branch used the fact that WC Dextrin is effective in preventing destruction of dye by overreduction. This information was relayed to our Central Research Laboratory for explanation. Ted Larsen, a physicist at Easton, proceeded to carry out an excellent examination of this data. By examining spectrophotometrically the progressive reduction of Vat Blue BC, he was able to verify the four reduction stages of this dye as given by Houben *(3)*. More important, he was able to determine the reduction potential required to bring about each of these stages. He found that its first stage of reduction occurred at about 860 ± 20 mv. He then turned to measure the caustic-hydro system. Here he found that, unbuffered, the system developed about 1200 mv at 190°F, while buffered with WC Dextrin, in amounts determined by practice, a potential of only 950 mv was developed. Thus, the WC Dextrin acted as a buffer in protecting Vat Blue BC from overreduction. With this phase of the problem answered, the practical measurement and control of reduction potential was taken up by the dyestuff-applications group. The Physics Department, notably F Maisch and J P Beiswanger, were very helpful in assembling equipment similar to that used by Larsen and in modifying it for wide flexibility of application. Our first attempts with small swatches in beakers were inconclusive, so we graduated to a large stainless-steel bucket and larger samples. By measuring the potential developed and then maintaining it with suitable additions of caustic, hydro and WC Dextrin, we were finally able to hold a bath containing 2 lb/gallon of Vat Blue BC in reduced form at 200°F for as long as 4 hours without decomposition. At this point we realized that further experiments of this type would contribute little, so we transferred our work to the laboratory Williams Unit which is supervised by J H Hennessey of G D C. After controlled dyeing of about 4000 yards of four-inch material with Vat Blue BC by the dextrin method, we were advised of the use of sodium nitrite as a protectant in the industry. Our investigation with nitrite showed that it offered advantages over dextrin if it could be controlled. Reports from industry complained of excessive consumption of hydro when nitrite was employed. Further study of this reaction led to the discovery that ammonia was liberated from the unit during the run. The only source of ammonia had to be the nitrite. That is, in the presence of a strong reducing system, such as caustic and hydro, nitrite is converted to ammonia. Fortunately, the alkaline reduction of nitrite to ammonia has been carefully studied and the intermediate steps together with the potentials required to bring each one about are reported in the literature *(4)*.

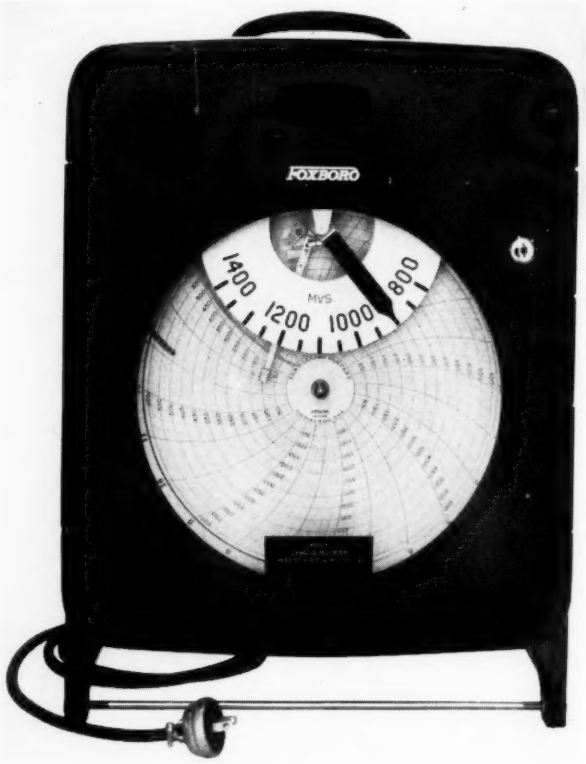

Figure 1

Examination of these data show that nitrite is first reduced to hyponitrite, then hyponitrite to hydroxylamine, and finally hydroxylamine to ammonia, as shown in the following series, where the change in the charge on the nitrogen is from +3 to −3:

I) +3 II) +1 III) −1 IV) −3

$$NO_2^- \xrightarrow[180\text{ mv}]{} N_2O_2^{=} \xrightarrow[730\text{ mv}]{} NH_2OH \xrightarrow[-420\text{ mv}]{} NH_3$$

This means that, to go from I to II, one need only provide a reduction potential in excess of 180 mv, from II to III a potential in excess of 730 mv, but from III to IV the reaction is spontaneous as indicated by the negative 420. This step therefore rapidly consumes reduction potential. Step 2 to 3 with its 730 mv is the buffering step as far as dye is concerned. It presents a sufficiently high potential to prevent rapid consumption of chemicals but not as high as the dye, so that it will preferentially consume a portion of the potential developed. This protects the dye. Unfortunately, step III to IV does occur and consumes our reduction potential rapidly. If we include in our bath sufficient nitrite to protect from the very beginning, then we will generate so much hydroxylamine that it will consume the major portion of our hydro. This would account for the reports from the trade of excessive consumption of hydro. Logically, one should anticipate the development of this hydroxylamine, include an estimate of the equilibrium amounts in the starting bath and then include sufficient nitrite to protect the dye from the remaining potential. This is what has been successfully done. With this picture in mind, we returned to the laboratory Williams Unit and processed some 6000 yards of 4-inch material using a combination of nitrite and hydroxylamine in the unit charge and feeding only nitrite during the runs. By this time we had established the following method of operation: if the potential dropped, we first made an addition of hydro because we realized that this material was most likely to disappear first. If this addition did not immediately cause a rise in the potential, a deficiency in caustic was indicated. We then would make additions accordingly, keeping in mind the simple fundamental that the total amount of hydrosulfite added to a dyebath must not much exceed the weight of caustic soda calculated as

flakes.

After we had established what we considered were optimum ratios, J H Hennessey arranged for the first of many practical runs. The fabric was an English broadcloth to be padded and wet-developed at 200° F in the Williams Unit. The shade was a royal blue (1 lb/gallon Vat Blue BC Single Paste); 2000 yards were dyed with control. The bath was charged with hydroxylamine and nitrite in addition to caustic, hydro and color. We were able to maintain a constant potential at 925 mv by suitable addition of nitrite, caustic and hydro. The dyeing was excellent.

The second run was a shirting shade (1.6 oz/gallon Vat Blue BC). This run of 7000 yards was padded and wet developed in the Williams Unit at 205° F. A potential of 925-950 mv was maintained throughout the run. Shading from end to end was excellent. Here again, the nitrite-hydroxylamine system was employed.

At this point we felt that the process was practical but two questions remained unanswered. Could we control for long periods of time without difficulty, and could we control Vat Blue BC beyond that critical 7000-yard mark?

Our next trial was directed toward answering the first of these questions. We controlled the Williams Unit/water-phase development of an OD #7 shade on #10 Duck for a period of 24 hours by addition of caustic and hydro. During this time over 100,000 yards was processed. The titrations made during this period indicated as follows:—

caustic soda, 2.4-3.5 oz/gal.
hydrosulfite, 1.28-2.54 oz/gal.

Despite these variations the potential developed was 1100 ± 5 mv. At one point during the run the potential dropped sharply. Investigation showed a failure in the caustic feed system. This would not have been detected for at least 7 minutes by the titrations, during which time the reducing baths would have been lost, as well as 490 yards of goods. The dyer indicated that this one point constituted a saving to him of at least $500.00. Incidentally, the machine dyer alone with a few words of explanation maintained a correct potential for 16 of these 24 hours.

The second question, that of control of Vat Blue BC, was answered at another mill. Here we were able to control by the nitrite process the development of a 4 oz/gal shade in a water-phase Williams Unit at 200° F. We ran 20,000 yards on one occasion and 35,000 yards of the same shade on another. The average consumption of chemicals for this development was as follows:

4.5 lb hydro/1000 yards
7.5 lb caustic soda (50% liquid)/1000 yards
0.3 lb nitrite/1000 yards

The potential was maintained at 925-945 mv throughout both runs. The results, in their opinion, were quite good. The coverage was considerably smoother than they had been able to obtain by development with the hot-oil process. Samples, taken every 1000 yards during the second run, are available here this evening for examination of uniformity of color.

The importance of being able to measure this potential was illustrated by an incident during the first run. A leaky steam valve caused the units to boil. This in turn caused the shade to go red and flat. Simultaneously, the rise in potential was noted and corrective measures in additions were made. By the time the dyer had decided to run in a leader and remake the units, the shade was normal again, and so these expensive measures were not necessary. It is interesting to note that titrations for hydro during 90% of these two runs indicated no excess present.

We have also checked a yellow shade dyed with 12 oz/gallon Vat Yellow, which was being developed in the Williams unit at 190° F. A potential of 975 mv was indicated by the concentrations which they currently employed. We recommend that they increase the feeds to give a potential of 1025 mv. This was done, and we controlled approximately 10,000 yards of this shade. The increase in potential necessitated a 10% cut in their pad formula.

A similar incident was experienced in measuring the development in a Williams Unit of OG #107 on 21,000 yards of 9-oz sateen, running at a speed of 115 yd/minute. Here an initial potential of 1000 mv was increased to 1100 mv by an increase in hydro feed. The caustic was determined as excessive to begin with, so its feed was not changed. This change necessitated a 5% decrease in pad formula.

These examples will serve to illustrate the type of control that we are able to maintain, the economies which may result and, perhaps most important, the excellent quality that may be expected in controlled continuous water-phase development of vat dyes. We have at the moment controlled the dyeing of some 300,000 yards of material in water-phase dyeing in Williams Units.

PAD STEAM AND HOT OIL——The pad-steam method and hot-oil method are similar in that both require an impregnation with caustic soda and hydrosulfite prior to actual development. This chemical pad is normally maintained at relatively low temperatures (90-115° F) in order to avoid excessive decomposition of chemicals. It must not be maintained much lower than this because of excessive cooling of either steam or hot oil, as the case may be. The system of caustic and hydro, even at these low temperatures, develops a measurable potential, and, conseqeuntly, a control of the concentrations in this chemical pad is possible by correct interpretation of reduction-potential measurements. Contrary to the water-phase development previously discussed, a titratable excess of caustic and hydro are necessary in the chemical pad because the intent here is to impregnate for subsequent reduction and not to reduce completely at once. Therefore, the value of titrations here is considerably greater, and reduction-potential measurements are not necessarily more accurate. It should be pointed out, however, that these measurements are instantaneous and do not require the expense of someone to titrate. Our practical trials to date have shown that these two factors are definitely advantageous to the dyer.

We realize that there are other continuous methods for vat dyeing, but, unfortunately, we have not yet had an opportunity to investigate them. We do feel that reduction-potential measurement and control will also contribute to these methods.

BATCH DYEING——The last portion of this paper deals with vat dyeing by batch methods. This includes jig dyeing, jig development and package-machine work.

The introduction of reduction-potential control into jig dyeing was brought about by the problem of dyeing Fortisan, a highly oriented rayon. Because some difficulty had been encountered with this fabric, it was felt that the most efficiency would be realized if the dyebath was maintained constant at a maximum potential commensurate with the alkali sensitivity of this material. Many thousands of yards of Fortisan were dyed with this control and we found that the dyeing efficiency was improved sufficiently to enable us to cut the number of ends in dye by half in certain cases.

This work led naturally to the measurement of jig development work. Here again, as might be expected, the proper balance and control of caustic and hydro has brought about surprising results. Among the runs, which total about 20,000 yards, we observed that some of the jig baths were being operated with chemical concentrations far in excess of that actually required. Cuts in hydro consumption of as much as 6 lb for a 1000-yard roll have been made. In other cases, definite deficiencies were indicated. One plant was able to cut the pad formula 20% as a result of increasing the caustic concentration by 30%. In another case, involv-

ing a Sea Island poplin, the amount of caustic had to be increased from 14 to 26 quarts, as indicated by the instrument, before a satisfactory dyeing was obtained. Very seldom did the recording of the potential developed indicate that optimum conditions existed; for the most part these measurements have shown that some adjustment in the chemical balance was necessary.

Our work on package-machine dyeing has been very limited. The results that we have obtained to date strongly indicate that more consistent results will be obtained when proper balance, as indicated by reduction-potential measurements, are established.

In one practical run the measurements indicated definite excesses of chemicals. Another indicated a deficiency of hydro. The correction of this deficiency brought about an increase in color value in the run.

AUTOMATIC FEED CONTROL——In this paper I have neglected completely any discussion of automatic feed control. This problem lies entirely within the domain of the instrument manufacturer. They have assured us that such automatic feed control is quite possible.

OTHER USES——This type of equipment has many ramifications for the wet processor. A few of these are the determination of purity of hydro, the measurement and control of sulfur-color application and the measurement and control of oxidation. This latter would, of course, cover bleaching operations. We feel that these and many other applications will be explored and utilized once their importance is established.

In conclusion, I would like to thank the many persons who contributed to the development of this process, the research men, the demonstrators, the mill men and the salesmen. In particular, I would like to acknowledge the contributions of our dye application research group composed of E L Youse and D Eicklin during early development and the application of the practical know-how of J H Hennessey in vat dyeing throughout the development of this process.

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Meeting Report— New York Section

April 18, 1952
Kohler's Swiss Chalet, Rochelle Park, N J

A MEETING of the New York Section was held on Friday evening, April 18, 1952 at Kohler's Swiss Chalet, Rochelle Park, New Jersey. A social hour and dinner preceded the meeting.

The Chairman, Paul J Luck, introduced the following guests: R Hobart Souther, Chairman of the Piedmont Section, Rudolph C Geering, Chairman of the Western New England Section, Dr George Wham, Director of Research at the University of Texas, Dr Harold W Stiegler, AATCC Director of Research, and Robert Burns of Celanese Corporation of America.

The Chairman announced that the date of the Annual Outing had been changed from June 20th to June 13th and would be held at the North Jersey Country Club. Ernest Gsell, Outing Chairman, will report on plans at the May meeting.

A letter from Ernest R Kaswell, Chairman of the Technical Program Committee for the 1952 National Convention in Boston, inviting members to contribute papers, was read.

The Chairman announced that John H Hennessey, Treasurer of the 1951 National Convention, had presented a check at the National Council meeting that day in the amount of $21,306.52 to be turned over to the research fund.

It was announced that the next meeting would be held on May 16th at the Swiss Chalet to celebrate the 30th Anniversary of the founding of the Section. Thirteen past chairmen of the Section, who are still members of the Association, would be invited as guests.

Frederick Fortess, Section Head, Textile Lubrication, Sizing and Finishing Dept, Central Research Laboratories, Celanese Corporation of America discussed "Some Aspects of Fabric Finishing". Daniel Sassi, Research Laboratory, Interchemical Corporation, presented a paper entitled "Rapid Method of Fiber Identification in Blends". Both papers were illustrated by slides.

The attendance was approximately 250.

Respectfully submitted,
NORMAN A JOHNSON, *Secretary*

—♦ ♦—

NNE Section Meets With LTI Student Chapter

MEMBERS of the Northern New England Section were treated to three papers by members of the Lowell Textile Institute Student Chapter at the April 18th meeting of the two groups at Lowell.

Three movies were shown prior to presentation of the papers, including "Cross Roads for America", which dramatized the need for labor-management cooperation; "Metachrome Dyeing with Metomega Chrome Colors"; and "The Price of Freedom", the broad theme of which was the responsibility of leadership.

Brief abstracts of the student papers follow:

"THE CURING OF UREA-FORMALDEHYDE RESINS BY CONVECTION & INFRARED METHODS"—Samuel Wood (Speaker), Ruth Denio

The polymerization of urea-formaldehyde resins was studied in solution by measuring the change in opacity of the resin films as they were cured. Then the resin was applied to a cotton fabric and the physical properties of the fabric used as a measure of the cure. On the basis of the results, it was concluded that a cure by infra-red was comparable to one by convectional methods. Qualitative measurements of factors affecting the speed of polymerization were also made.

"SOME ASPECTS OF A NEW SOLVENT EXTRACTION OF RAW WOOL" —Florence Liston (Speaker), Milton Boches

This paper discussed the possibilities of a new system for scouring raw wool using methyl and isopropyl alcohol. It was shown that the wax recovered from this process is of better quality than that wax now being recovered from conventional methods. A comparison of the felting of wool by the normal soap and soda scouring and the alcohol system was discussed showing that the felting of wool scoured in the alcohol was far less than that felting attained in soap and soda scouring.

"THE MECHANICAL PROPERTIES OF MELAMINE - FORMALDEHYDE TREATED VISCOSE RAYON YARNS" —Alfred L Cate (Speaker), Donald P Feyler, Fred B Shippee

In this study, a method was demonstrated for determining at a low elongation, the mechanical properties of the warp yarns of melamine-formaldehyde resin treated viscose fabrics. The changes produced in the mechanical properties of the yarn with a change in the resin content were shown.

It was demonstrated by means of the three components of yarn elongation that an increase in the resin resulted in the increase in the immediate elastic recovery as a result of a loss of some of the permanent set, since the delayed recovery remained constant. The changes produced in the crimp were indicated, also.

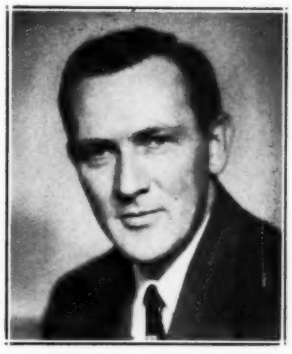
30th Annual Convention
Finishing Group——

ADVANCES IN THEORETICAL AND PRACTICAL STUDIES OF GAS FADING*

V S SALVIN, W D PAIST & W J MYLES
Celanese Corporation of America, Summit, N J

V S Salvin

W D Paist



W J Myles

This paper describes the phenomenon of gas fading and discusses the mechanism of the reaction in relation to the nature of the fabric, as well as the use of newer dyes and inhibitors. The gas fading of dyed acetate fabrics is due to nitrogen dioxide, which is absorbed from the atmosphere. This gas is very soluble in cellulose acetate but does not react with the fiber to any extensive degree. Certain other fibers gas-fade like acetate. Data are presented regarding the absorption of nitrogen dioxide by cotton and nylon. The acetyl value of the fiber, as well as its moisture content, has a marked effect on the rate of gas fading. It is found that the rate of gas fading can be correlated with dye structure. The nature of the chemical changes in dyestuffs and advances in the synthesis of resistant dye structures are discussed. The type of chemical change is of importance in evaluating the newer dyes in accelerated tests. The application and effectiveness of permanent and fugitive inhibitors is reviewed, and an attempt is made to correlate inhibitor structures and properties.

INTRODUCTION

GAS fading is the fading of dyed fabric by acidic gases in the atmosphere, which are formed in combustion processes. It was shown by Rowe and Chamberlain in 1937 *(5)* that the active constituents of the combustion gases are nitrogen oxides. These are normally present in air to the extent of about 2 parts per hundred million. A recent study of the atmosphere over Los Angeles *(36)* showed nitrogen oxides are present at 2 parts per 10 million on a typical smoggy day. It was calculated that approximately 284 tons of nitrogen oxides are liberated into the atmosphere of Los Angeles County each day by various combustion processes. The undiluted products of the combustion of illuminating gas contain about 2 parts per ten thousand of nitrogen oxides *(2)*. The present standard AATCC method for the rapid evaluation of the fastness of dyed fabrics is based on the action of such spent gases *(8)*. The concentration of nitrogen oxides in this chamber is in the vicinity of 2 parts per hundred thousand.

As was emphasized by Henry Dreyfus in the Perkin Medal Address in 1939 *(6)*, it is the blue, violet, and red aminoanthraquinone dyes that are most vulnerable to gas fading. Certain azo acetate dyes are also not completely resistant to gas fading. This has caused considerable difficulty in producing a full range of dyes for cellulose acetate. The situation is much improved at the present time.

These same vulnerable dyes do not fade on wool, nylon, or Orlon, but they do fade on Dacron polyester fiber. In practice, the aminoanthraquinones are not used on cotton or viscose but if they are applied to these fibers by vatting, they fade at a very slow rate. It should therefore be stressed that the fiber as well as the dye plays a part in gas fading.

EXPERIMENTAL

THE CAUSE OF GAS FADING——It is of some importance to know which of the several nitrogen oxides is responsible for gas fading. Ten years ago Greenspan and Spoerri *(17)* reported that nitric oxide gave a color change with dyed acetate most nearly like that occurring during normal gas fading. The pure gas was used in their experiment. However, the presence of only traces of oxygen remaining in the fabric or in the walls of the apparatus would have been sufficient to form enough nitrogen dioxide to cause the slight color change which was observed. Other workers have since shown that nitrogen dioxide, when generated in concentrations of one part per thousand or less, will give color changes approximating those caused by combustion gases. Since nitric oxide in the presence of a large excess of oxygen is rapidly oxidized to nitrogen dioxide, it seems probable that it is the latter material which is primarily responsible for gas fading. It is not known whether it is the gaseous nitrogen dioxide or its hydrolysis products which normally exist and react within the cellulose acetate. It may be of interest that Hughes, Ingold and Ridd *(46)* have recently held that the diazotization of aromatic amines in weakly acid solutions of nitrous acid (a condition which resembles that in cellulose acetate during gas fading) is a reaction between the free amine and

* Presented by W J Myles at the Hotel Statler in New York, N Y, on October 19, 1951.

TABLE I

RELATIVE AMOUNTS OF NITROGEN DIOXIDE LIBERATED FROM EXPOSED FABRICS (DRY-CONTACT METHOD)

	Change of Test Fabric With Contact Time	
Undyed Fabric	*30 Minutes*	*60 Minutes*
cotton	no change	no change
viscose	no change	no change
CELCOS	slight fading	moderate fading
acetate, bright	moderate fading	bad fading
DACRON	moderate fading	bad fading
wool	no change	very slight fading
nylon	no change	no change

N_2O_3 formed by decomposition of the nitrous acid.

Some idea of the difference in behavior between nitric oxide and nitrogen dioxide can be gained from a simple experiment on a chloroform solution of Celanthrene Blue FFS. One hundred ml of dry chloroform containing 20 mg of the dye was put in a 230-ml bottle. Nitrogen was bubbled through the solution for 30 minutes. The bottle was evacuated until the chloroform had boiled gently for a minute, then filled through a two-way cock with nitric oxide. After several hours, there was a barely perceptible reddening of the color, and no further change in 48 hours. Finally, 2 ml of nitrogen dioxide gas was injected into this bottle, and after several hours a slow fading was perceptible. Two ml of nitrogen dioxide gas injected into a similar bottle caused complete fading in 10 minutes. Under the conditions described, nitric oxide appears to inhibit gas fading instead of causing it.

THE ABSORPTION OF NITROGEN DIOXIDE BY VARIOUS FIBERS——It is known that nitrogen dioxide is absorbed by cellulose acetate and remains in large part unchanged *(17, 30)*. For example, acetate fabric may be exposed for one hour at room temperatures to nitrogen dioxide generated in the standard AATCC chamber by the addition of 50 ml of 5% phosphoric acid to 3 grams of sodium nitrite in 50 ml of water. After a ten-minute airing, if the exposed fabric is pressed for one hour onto a strip of the AATCC Standard Test Fabric #1, the test fabric will be considerably faded. This test, applied to various fabrics, gave the results shown in Table I.

In a second test method, a 2" x 2" piece of the exposed fabric is placed for 1 hour at room temperature in 50 ml of water made acid with 1 ml of acetic acid and 5 ml of 1% hydrochloric acid. Nitrogen dioxide diffusing into the water will cause the fading of a ½" x 1" piece of the AATCC test fabric. The results of this test on several fabrics are shown in Table II.

It is shown by the dry-contact method that acetate and Dacron liberate considerable quantities of nitrogen dioxide, which would normally be effective in fading the dyes. Nylon and viscose do not liberate any gas. However, the aqueous-transfer method shows that gas absorption by the nylon has nevertheless occurred, because fading of the test piece takes place under these conditions. It is believed that the gas is held in the nylon by salt formation, or more probably by nitrosation of the amide links in the polymer structure, and that this compound is broken up by the acid in the aqueous test. Likewise, fabric padded with melamine or triethanolamine and exposed to nitrogen dioxide does not cause fading in the dry-contact test, but does release nitrous acid in the aqueous-transfer test.

TABLE II

RELATIVE AMOUNTS OF NITROGEN DIOXIDE LIBERATED FROM EXPOSED FABRICS (AQUEOUS TRANSFER METHOD)

Exposed Fabric	*Change of Test Fabric*
none	practically no change
cotton	practically no change
viscose	practically no change
CELCOS	slight to moderate reddening
acetate, bright	moderate reddening
wool	very slight reddening
nylon	slight reddening
ORLON	practically no change
DACRON	slight reddening

Viscose and cotton do not present as clear a picture. It is possible that nitrogen dioxide is absorbed by these fabrics and reacts with some part of the cellulose structure. Experiments at several degrees of acetylation show that, as the substrate becomes more like cellulose, the rate of gas fading decreases and moisture absorption and viscose dye affinity increase. On the other hand, the addition of alcohols, such as ethylene glycol or glycerin, to cellulose acetate does not materially interfere with the rate of gas fading. The fate of the nitrogen dioxide absorbed in cellulose under gas-fading conditions is still a matter for speculation. Whatever the reaction, it does not measurably change the physical properties of the fabric. Viscose and acetate satins exposed to five units of gas fading (equivalent to 3-4 years of service life) showed no significant change in tensile strength or elongation. Likewise, tests on acetates badly faded in service conditions showed no changes in acetyl value, viscosity, or tensile strength because of gas fading.

A vivid idea of the relative rates of absorption of nitrogen dioxide by various fabrics can be obtained by putting a 6-gram piece of the fabric in a 2-liter flask containing 6% nitrogen dioxide. Using specimens conditioned at 50% RH, it is seen that nylon absorbs all the red-brown gas in ten minutes. Acetate and viscose absorb fairly rapidly, but some gas is still present after several hours. When finally the excess gas is blown out, the acetate soon releases a large amount of gas, while a trace is released by the viscose, and none at all by the nylon. Bone-dry viscose does not absorb nitrogen dioxide; dry acetate absorbs, but much less than at 50% RH. In a similar test on 3-gram samples of acetate films at 50% RH, it was found that gas absorption increased with decreasing acetyl value. Visual estimates of the relative absorption are shown in Table III.

TABLE III

EFFECT OF ACETYL VALUE ON ABSORPTION OF NO_2

% Combined Acetic Acid	*Relative Absorption*
Blank	0.00
59.8	.83
62.0	.85
54.5	.88
49.6(a)	.89
49.6(b)	.91
41.3	.95

When, after 53 hours, the flasks were cleared of excess NO_2 and restoppered, it was found that most NO_2 was released by the film of 59.7% acetyl value, least by the film of 41.3% acetyl value. Thus, the film which had absorbed the most gas (and in which gas fading occurs most slowly) gave up the least, and the film which had absorbed the least gas (and in which gas fading occurs much more rapidly) gave up the most. It must be stressed, however, that the gas in these tests is a great deal more concentrated than under normal gas-fading conditions, and the rates as well as the character of reactions may be rather different at very low concentrations.

The effect of moisture and acetyl value on rate of gas fading was shown by exposing a series of dope-dyed films to one part per thousand of nitrogen dioxide in an apparatus to be described below. Results are shown in Table IV.

The results on dry films show that fading occurs more and more slowly as the acetyl value is reduced, and especially slowly below 50% acetic acid. The effect of moisture is to increase greatly the rate of fading, and to reduce the differences in rate shown at the several acetyl values.

TABLE IV
EFFECT OF ACETYL VALUE AND HUMIDITY ON RATE OF GAS FADING IN FILMS

% Combined Acetic Acid	Minutes to Fade 50%	
	0% R H	50% R H
61	44	12.5
60	44	9.7
58.7	53	10.0
58.5	56	12.0
56.2	67	12.0
54.5	68	11.6
49.6	150	31.5
45.8	435	47
54.5 + 17% diethyl phthalate	30	5.7

At 100% RH the fading time is further reduced to about one-fifth that at 50% RH. It is also noteworthy that the addition of 17% plasticizer to one film doubled its rate of fading.

Experiments are under way at present to measure the absorption of NO_2 by acetate films at concentrations of 1 part of NO_2 in 1000 of air, or less. Results thus far indicate that at 50% relative humidity absorption occurs rapidly, and the rates are about the same for samples having acetyl values of 44-60% combined acetic acid. Under the same conditions, dyed films of this acetyl range show a marked difference in their rates of fading. At 65% relative humidity, the lower acetyl material absorbs somewhat more rapidly than the higher. At both humidities, regenerated-cellulose film absorbs NO_2 at a rate only slightly less than does cellulose acetate. The resistance to gas fading of dyes in fibers of low acetyl value cannot be satisfactorily explained on the basis of a lower rate of NO_2 diffusion. It is possible that, under normal gas fading conditions, i e, at NO_2 concentrations of one part per ten million, the greater resistance in the low acetyl fabrics is due to a combination of (a) a lower rate of NO_2 diffusion, (b) a higher rate of reaction of the fiber with the gas, and (c) a lower rate of reaction of the dye with NO_2 in the medium of lower acetyl value.

The general conclusion to be drawn from these experiments is that the troublesome gas fading of aminoanthraquinones in cellulose acetate is due to the relatively great solubility of nitrogen dioxide in this material and the low rate of reaction with it. The gas is therefore free to diffuse through the fibers and to attack the dyes.

GAS-FADING INHIBITORS —— Gas fading can be reduced by partial saponification of the fabric, or by covering the fibers with a less permeable polymer coating, but both of these procedures are usually unacceptable from a practical point of view. A better method is to treat the fabric with some compound that combines with the nitrogen dioxide (or nitrous acid, etc) before it can fade the dye. Inhibitors currently in use are of two types. The fugitive inhibitors, such as melamine, triethanolamine, or sodium formate, are applied in a finishing treatment. They give reasonable protection, depending on the depth of shade and the sensitivity of the dye. Their protection is reduced by perspiration and lost on washing. Some compounds, such as triethanolamine, reduce the light fastness of viscose dyes, and their use presents problems with viscose-acetate blends. This type of inhibitor acts by maintaining an alkaline condition on the fabric, under which reactions of gas fading do not occur.

A second type of inhibitor has been developed in recent years. This is the permanent, substantive type, which can be applied during dyeing, represented by N,N'-diphenylethylene diamine. These are weakly basic aromatic amines, which act by forming nitrosamines and ring-nitrosated compounds. These latter are generally rather yellow, and the use of these inhibitors is accompanied by a yellow discoloration. This is not too objectionable where the quantity of inhibitor is adjusted to the depth of the shade. Inhibitors of discoloring type are not useful in pastel shades, since inhibitor discoloration can be just as objectionable as gas-fading change. These inhibitors also affect the light fastness of certain acetate dyestuffs.

The shortcomings of these several inhibitors have led to a search for new structures, which would be substantive and nondiscoloring. A consideration of the mechanisms by which nitrogen dioxide or nitrous acid may be consumed shows four major types:

1) The inhibitor may form a salt with nitrous or nitric acid (or other acids that catalyze the fading). The greater their intrinsic basicity, the greater will be the buffering action of these compounds.

2) The inhibitor may react to form a nitrosamine or a diazonium salt with the nitrogen dioxide by the same type of reaction which causes the dyes to fade.

3) A tertiary amine may react through cleavage of an alkyl group. This involves the oxidation of the alkyl group, reduction of NO_2 to NO, and formation of a nitrosamine.

4) The inhibitor may act as an antioxidant because of the ease with which NO_2 or nitrous acid are reduced to nitric oxide.

Examples of nondiscoloring substantive inhibitors are diphenylacetamidine and the aralkylamines. They are strong bases. Diphenylacetamidine neutralizes nitrous acid. It does not react with nitrous acid under acidic conditions. An equal weight of diphenylethylene diamine is more effective than diphenylacetamidine, because it reacts selectively with nitrous acid even in the presence of other acids to form the dinitrosoamine.

Using dyestuffs of varying degrees of gas-fading resistance, and inhibitors of both permanent and fugitive types, the degree of change on gas-fading exposure was determined. Both light and medium dyeings were examined. The dyestuffs included Celanthrene Blue FFS, Artisil Blue GLF, Amacel Blue GLF, and Eastman Blue GLF. The codyeing inhibitors used were diphenylethylene diamine codyed in 0.6% concentration, diphenylacetamidine in 1% concentration. Fugitive inhibitor used was melamine padded at 15 grams per liter with 100% wet pick-up.

Fabrics were exposed for 1, 2, and 3 AATCC gas-fading units. Color changes were noted by spectrophotometric, "Color-Eye", and visual observations. The results were checked by use of mixed dyeings in light shades.

Spectrophotometric curves bear out the observation that dyestuffs undergoing pronounced change in tone on gas fading, such as Celanthrene Blue FFS, which goes from blue to red, give most pronounced visual changes. Artisil Blue GLF retains the tone but loses moderately in depth. Amacel Blue GLF loses moderately in depth and goes slightly redder, whereas Eastman Blue GLF shows least reduction in depth and retains its tone.

Spectrophotometric curves further show that dyestuff changes are taking place concurrently with inhibitor change, as in the case of diphenylethylene diamine.

Mixed shades are most effective in showing relative resistance to gas fading. Thus, Artisil Blue GLF appears more resistant to gas fading in self shades than in mixed dyeings with resistant yellows and reds. Where a gas-fading red, such as the conventional 1-amino-4-hydroxy anthraquinone is used, the change observed is a combination change of the blue and the red.

The nondiscoloring substantive inhibitors, such as diphenylacetamidine, have potentialities for use in the lighter shades of the improved dyestuffs Amacel Blue GLF and Artisil Blue GLF. In these depths, the discoloration imparted by exposed diphenylethylene diamine negates its protective value and prevents the use of a discoloring inhibitor. The nondiscoloring inhibitors are not of great value on the dyestuffs of poor gas-fading resistance, such as the prototypes of Celanthrene Blue FFS. Since Eastman Blue GLF only shows slight gas-fading change, improvement from the use of diphenylacetamidine was not easily visible, but could be picked up on spectrophotometric curves and the "Color Eye".

The discoloring substantive inhibitors showed good protective value in the medium shades of Amacel Blue GLF, Artisil

Blue GLF, and Celanthrene Blue FFS. Observations at 2- and 3-unit exposures showed that the Amacel Blue and the Artisil Blue changed less than the Celanthrene Blue. As in the case of diphenylacetamidine, the diphenylethylene diamine did not give visual improvement for Eastman Blue GLF.

Melamine applications were more effective than either of the codyed inhibitors in giving protection to the dyeings. Combination of substantive inhibitor and fugitive inhibitor gives additive effects.

In exposures of 2 and 3 AATCC units, the advantages of using dyestuffs of intrinsically better gas-fading resistance were apparent, both with and without inhibitor.

A rapid and useful method for the evaluation of inhibitors in dope-dyed films was developed as follows: Films were prepared containing 0.4% Celanthrene Brilliant Blue FFS. The compound to be evaluated may also be added to the dope, usually at 2% on the acetate. A strip of film is cut 1 inch wide and 18 inches long. This is conditioned at 50% RH and inserted through a slit in a rubber gasket, which fits on a 20-liter wide-mouth jug. In the jug is placed a small dish containing 5 ml of 2% sodium nitrite. Five ml of 6% phosphoric acid is then added, and the film and gasket quickly set in place. The concentration of gas developed in the jug is about 1 part per thousand. At intervals a sample is clipped off the film strip. It is then baked 2 hours at 100° C. This is done to accelerate the fading by unreacted gas that is present in the film. The film color would otherwise not be stable. Finally, optical densities at 620 mμ are measured with a Lumetron colorimeter, and the results are plotted. The colors can be corrected to a standard thickness of 2 mils by taking advantage of the fact that fading proceeds toward the center of the film with a fairly sharp boundary between faded and unfaded dye. Six film strips are faded simultaneously, and the time for a standard amount of fading in a sample is compared with the time for an equal fading of an untreated control (or still better, with the time for a film containing a standard inhibitor). The ratio of fading times is called the "activity" of the inhibitor at a specified concentration. An initial screening can thus be carried out on 0.25 gram or less of inhibitor, and one is sure that it is actually present, evenly distributed, in a predetermined concentration. Table V gives the results of the evaluation by this method of a selected group of compounds.

TABLE V
INHIBITOR ACTIVITY BY FILM TECHNIQUE

Inhibitor (2%)	*Activity*	*Remarks*
none	1.0	
triethanolamine	4.1	water-soluble
dibenzylamine	2.5	not wash-fast
dibenzylethylene diamine	3.7	low affinity
tetrabenzylmethylene diamine	3.2	low affinity
tetrabenzylethylene diamine	2.9	no affinity
diethylaminoethyl phthalate	5.3	not wash-fast
α-diethylaminoacetanilide	2.9	low affinity
N,N′-diphenylacetamidine	3.0	fair affinity
N,N′-diphenylformamidine	4.5	low affinity; discol
2,4,5-triphenyl imidazoline	2.7	fair affinity
N,N′-diphenylethylene diamine	3.6	cons yellowing
N,N′-di(2,6-dimethyl) phenylethylene diamine	2.6	nondiscoloring
N,N′-diphenylpiperazine	3.0	cons yellowing
aniline	4.2	bad yellowing
urea	1.5	
thiourea	2.8	
N-allyl urea	1.7	
N-allyl thiourea	5.6	water-soluble
s-diphenyl thiourea	3.2	bad discoloring
s-diphenyl guanidine	4.7	sl discol; no affin
2-hexyl pyridine	1.6	odor
dibenzylcyanamide	1.1	
ethyl anthranilate	2.5	nondiscol; sl affin
benzoyl acetonitrile	1.5	
diethyl malonate	1.6	
diphenyl methane	1.6	
α-benzilmonoxime	2.4	
di-t-butyl-p-cresol	1.4	
diethylene glycol (4%)	1.1	
cobaltous acetate (0.36%)	1.5	

It is seen in this table that the aromatic amines, which show high substantivity and activity, suffer from a yellow discoloration. The same is true of compounds with phenolic or aromatic alkoxyl groups. Diphenylacetamidine, on the other hand, is a model inhibitor. If a similar compound could be prepared having greater substantivity and greater activity, most of the inhibitor problem would have been solved.

DYE STRUCTURE AND GAS FADING——Since the work of Rowe and Chamberlain *(5)*, it has been known that the main reaction of the dyestuff in gas fading is the diazotization of primary amino groups or nitrosation of secondary amino groups. They also suggested that oxidation of the dye may occur. We have found, for example, that completely faded Celanthrene Blue FFS shows a fluorescence in ultraviolet radiation and an acid-base change in 0.1% sodium carbonate solution. Both of these phenomena suggest an oxidative hydrolysis to a hydroxyanthraquinone.

Accelerated tests with aqueous nitrous-acid solutions, or higher concentrations of gaseous nitrogen dioxide, generally give color changes, which parallel those in the standard AATCC chamber and in service tests. There are some exceptions, however, which suggest that different reactions may occur in the accelerated tests.

1) Celanthrene Blue FFS faded in the standard chamber shows a marked fluorescence in ultraviolet rays; this is not found when it is faded by high NO_2 concentrations or in aqueous nitrous acid.

2) Fabrics faded red by generated NO_2 or aqueous nitrous acid return partly toward the blue when exposed to sunlight, whereas the same fabrics faded in the standard chamber or in service do not show this effect.

3) With certain dyes, such as Amacel Blue GLF, fading occurs slowly on tone to a blue-grey, while in generated nitrogen dioxide fading is toward the red.

The blue aminoanthraquinone disperse dyes are known to have the best dyeing properties and the best light fastness. In order to overcome their deficiency of gas fading, the search has led toward structures containing amino groups of lower reactivity. One way to attain this is to put electronegative substituents in the positions ortho to the amino groups. An analogy is that between p-nitroaniline, which diazotizes readily, and 2,6-dichlor-p-nitroaniline, which does not. A second approach has been the synthesis of dyes containing only aryl amino groups, which are less vulnerable. Figure 1 shows the structures of some types which have been tested.

The gas-fading resistance of these dyes has been found to be inversely proportional to their ease of salt formation. This can be shown qualitatively by noting the concentration of hydrochloric acid that holds the dye in solution. Structure I in Figure 1 is easily soluble in 5% HCl, while Structure IV is not soluble even in 30% HCl.

A more elegant method of showing the difference in basicity of these dyestuffs is to dissolve them in glacial acetic acid and to titrate with perchloric in acetic acid. Figure 2 shows the titration curves obtained with several of these same dyes.

The titration curves show that Structures I and II are considerably stronger bases than Structures III and IV. The latter are much more resistant to gas fading than the former. The greater height of Curve IV, compared with Curve III, is

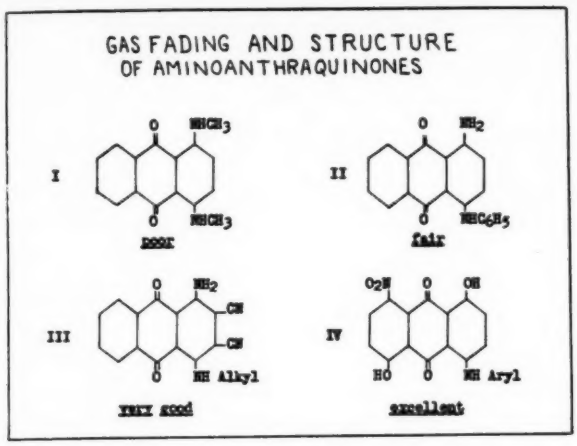


Figure 1

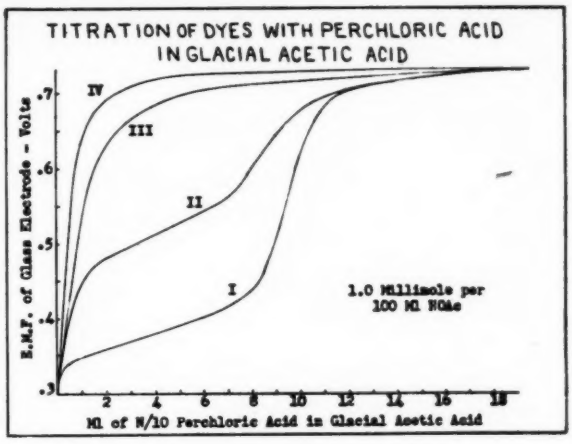


Figure 2

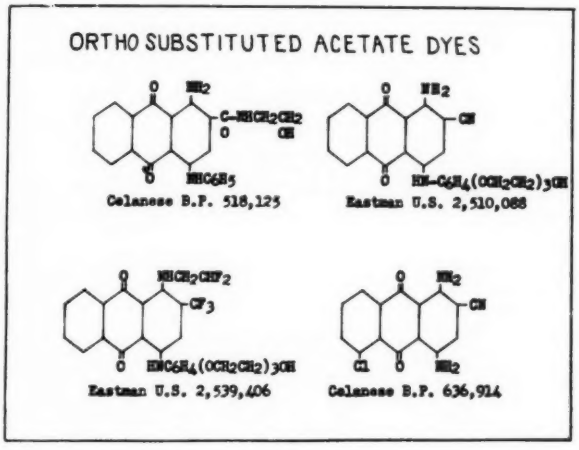


Figure 3

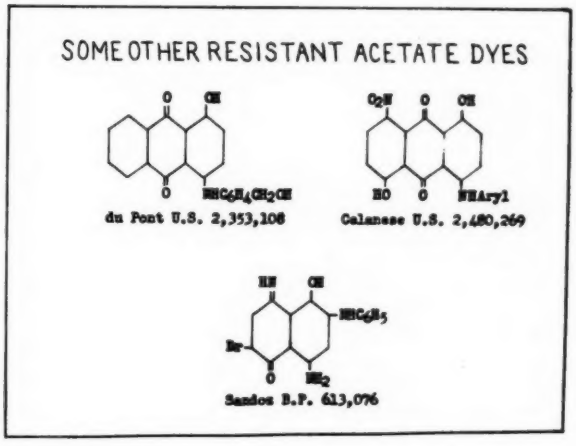


Figure 4

reflected in slightly better gas fading of Structure IV compared with Structure III. The same is true of the other two dyes.

A study of the patent literature on new acetate dyestuffs shows that the principles of ortho substitution and resistant arylamino groups are finding wide application. Figures 3 and 4 show the structure of a number of these dyes, which are said to be resistant to gas fading.

The search for improved blue acetate dyes has also led to a study of azo structures, naphthazarins, and developed naphthol dyes. Azo dyes, such as Setacyl Blue G and Eastone Blue BGF, are quite resistant, but show inferior light fastness. Attempts to improve light fastness with azo structures not containing ortho nitro groups have not been successful.

Blue dyestuffs with the naphthazarin structure have affinity for cellulose acetate. Their gas-fading resistance is superior to that of the conventional anthraquinone blues, and their blue tone is retained during fading. Sandoz has recently patented certain ortho-substituted naphthazarin blues which are said to have good gas-fading resistance (BP 613,076 & 653,356).

In the case of developed naphthol dyes, heavy blue shades with good fastness to light, washing, and gas fading can be produced on acetate. The coupling of Developer Blue BB with Naphthol AS-D gives a good blue if the development is carried out in the presence of 2% butyl carbitol or 1% of a nonionic, such as Alrosol C.

SUMMARY & CONCLUSIONS

The mechanism of gas fading on cellulose acetate is shown to be due to the relatively great absorption of nitrogen dioxide in this material and the low rate of reaction with it. The gas is therefore free to diffuse through the fibers and to attack vulnerable dyes.

Gas-fading inhibitors function by (a) maintenance of alkaline conditions on the fabric, or (b) selective reaction chemically with oxides of nitrogen to form nitroso compounds.

Correlation of gas-fading resistance has been made with dyestuff structure and with intrinsic basicity of the dyestuff. Dyestuffs of low intrinsic basicity show least gas-fading change.

The gas-fading resistance of the commercially available blue acetate dyes has been examined, and the usefulness of various inhibitors, both of the substantive and fugitive types, evaluated.

It is clear that the problem of increasing the gas-fading resistance of dyed acetate fabrics has received and is receiving considerable attention from both the yarn producers and the dye and textile-auxiliary manufacturers. The number of gas-fading complaints has been markedly reduced and will be reduced much further as the use of improved dyes, such as Artisil Blue GLF, Amacel Blue GLF, Eastman Blue GLF, and Celanthrene Violet BGF, increases. These dyes, in conjunction with improved inhibitors of both permanent

and fugitive types, can give fabrics with the required degree of color fastness and should contribute to the expansion of acetate fiber into new fields.

DISCUSSION

Question: Have you ever in your experience seen any gas fading of direct dyes on viscose?

Answer: Any gas-fading change that is obtained on viscose is probably due to sensitivity of the direct dye to acid like that exhibited by an acid-base indicator. As the direct dyes are not amino anthraquinones, they are not so susceptible to gas failure; but yet some changes in color are occasionally noticed.

Q: What are the chances of producing a fiber with some sort of inhibitor incorporated before spinning?

A: Considerable attention has been paid to the possibility of including inhibitors in cellulose acetate during manufacture. There are certain requirements, however, that are difficult to meet: 1) It must not affect the strength of the fiber, 2) it must be fast to washing and wet processing, 3) it must not change the dyeing and cross dyeing properties of acetate, that is, it must especially not increase the affinity for acid dyes, and 4) it must not reduce the light fastness of dyed acetate too much. Thus far a substance fulfilling these four requirements has not been found. Although the project of finding such an agent has not been abandoned, the discovery of new and better dyestuffs makes the problem less pressing.

Q: In terms of consumer use and experience, can you venture any figure as to how long a consumer might expect to have a garment and to use it satisfactorily before noticeable gas fading takes place?

A: As we are primarily interested in consumer satisfaction, we are looking for fastness to gas fading that will withstand ordinary use, regular dry cleaning and normal washing; fastness to gas fading on only the retailer's shelf is not enough. Now, to answer your question, according to our experience we do not get a complaint until a little more fading has taken place than that equivalent to two AATCC units of exposure.

If you have a drapery, fading takes place first on the outside of the folds and gradually toward the inside. If fading is on tone, the average consumer does not notice it readily, but if there is a change in shade, the customer becomes aware of it very soon and complains. The tendency has therefore been to get dyestuffs or inhibitors that give us gas fading in tone. Does that answer your question?

Q: Only partly, you have not covered the time element satisfactorily.

A: For suitings, I should think a customer would expect two seasons of use. Again I must emphasize that fading in tone is more important than the degree of fade.

Q: The two dyes in your experiments that stood up so well—are they available to the trade for trial?

A: They are now in pilot-plant production. Other companies are also active in developing new dyestuffs for acetate. I venture the prediction that those dyes will be fastest to gas fading that have the best light fastness on Dacron polyester fiber, and I therefore will be happy to see more work on Dacron dyestuffs.

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2,211,861	Aug 20, 1940	H Platt, R R Sitzler (Celanese): Dry heat, with or without benzylethylaniline, etc.
2,255,090	Sept 9, 1941	J M Tinker, D E Kvalnes (du Pont): Phenyl biguanide.
2,298,401	Oct 13, 1943	J G McNally, E C Jensen (Eastman Kodak): N-β-hydroxyethylethylenediamine, etc.
2,340,375	Feb 1, 1944	C H Giles, D G Wilkinson (Imperial Chemical Ind): 2-Methylbenzimidazole, amarine, etc. See also Brit 543,594.
2,366,241	Jan 2, 1945	C Dreyfus (Celanese): Partial saponification of fabric with conc alkali.
2,369,122	Feb 13, 1945	C H Giles, D G Wilkinson (Imperial Chemical Ind): N,N′-Diaryl formamidines or acetamidines.
2,371,221	Mar 13, 1945	R Burns (Celanese): Form an insoluble tannate on the fabric.
2,403,019	July 2, 1946	H C Olpin, S A Gibson (Brit Celanese): Padding with melamine-aldehyde resin.
2,409,257	Oct 15, 1946	C M Croft, T S Waller (Celanese): Padding with triethanolamine.
2,409,325	Oct 15, 1946	G C Ward, B T Pull (Celanese): Padding with amines, such as 3-amino-4-heptanol and cationic agents.
2,416,380	Feb 25, 1947	B Collie, C H Giles, D G Wilkinson (Imperial Chemical Ind): N,N′-Diphenylethylenediamine, etc. See also Brit 558,784.
2,440,330	Apr 27, 1948	H Dreyfus (Brit Celanese): 6% Cyanamide solution, dried and cured at 130°C.
2,482,710	Sept 20, 1949	D W Jayne, Jr, H M Day (American Cyanamid): Aniline-dicyandiamide reaction product.
2,495,810	Jan 31, 1950	W H Hindle (Celanese): Solvent swelling with slight saponification gives more resistant dyeings. See also Brit 583,349; 636,501.
2,518,393	Aug 8, 1950	F H Smith (du Pont): Stable emulsion pastes of N,N′-diphenylethylenediamine, etc.
2,525,898	Oct 17, 1950	H W Grimmel, A Guenther (General Aniline and Film): 1,3-Diphenylimidazolidine, etc.
2,529,016	Nov 7, 1950	H W Grimmel, J F Morgan (General Aniline and Film): N-Benzylaminotriazoles and tetrazoles. See also Brit 648,631.
2,529,935	Nov 14, 1950	H W Grimmel, A Guenther (General Aniline and Film): 1,4-Diarylpiperazines.
2,536,640	Jan 2, 1951	J J Hayden (General Aniline and Film): 1,1-bis-(p-aminophenyl) cyclohexane.
2,539,212	Jan 23, 1951	A F Strobel, W W Williams (General Aniline and Film): N,N′-Dibenzylethylenediamine, etc.
2,541,822	Feb 13, 1951	D E Kvalnes, C W Croco (du Pont): Disodium di-N-methanesulfonate of N,N′-diphenylethylene diamine.
2,546,167	Mar 27, 1951	V S Salvin, J R Adams, Jr (Celanese): Padding with an emulsion of N,N′-dibenzylethylenediamine.
2,546,168	Mar 27, 1951	W J Hart, V S Salvin (Celanese): Preparation of an emulsion for application of inhibitors.
2,546,169	Mar 27, 1951	V S Salvin, J R Adams, Jr (Celanese): Process for preparation of N,N′-di substituted imidazolidines in a dispersing medium.
2,567,130	Sept 4, 1951	F H Smith (du Pont): Water dispersible paste of N,N′-diphenylethylene diamine.

BRITISH PATENTS

243,841	Sept 23, 1924	G H Ellis (Celanese): Pad with aniline or alkylaniline. See also U S 1,723,230.
340,541	June 25, 1929	G H Ellis, T Ockman, H C Olpin (Celanese): Tetramethyldiaminodiphenylmethane. See also U S 1,930,232; Fr 694,751.
340,572	June 25, 1929	(Celanese): Alkyl or aryl derivatives of urea or thiourea, with or without borax.
355,726	Mar 21, 1930	G H Ellis, H C Olpin, E W Kirk (Celanese): Amides and N-substituted amides. See also U S 1,957,493.
361,362	May 15, 1930	G H Ellis, T Ockman, H C Olpin (Celanese): Benzylaniline or benzylethyl aniline.
361,381	May 15, 1930	G H Ellis, T Ockman, H C Olpin (Celanese): Benzylaniline, etc, used on animal or vegetable fibers.
373,628	May 20, 1932	(Amer Celanese): Thiourea, with or without borax. See also U S 1,972,111.
378,473	Aug 11, 1932	G H Ellis, E W Kirk (Celanese): Mono, di, tribenzylamine, octylamines, etc.
406,653	Feb 23, 1934	G H Ellis, E W Kirk (Celanese): Fixation of thiourea by steaming.
406,686	Feb 27, 1934	H C Olpin, G H Ellis (Celanese): α-Diethylaminoacetanilide, etc.
450,475	July 20, 1936	(I G Farben): Pad 30 minutes at 50°C with phenylthiosemicarbazide.
460,027	Jan 19, 1937	H M Bunbury, C H Giles (Imperial Chemical Ind): Pad with 5% sodium thiosulfate.
507,722	June 20, 1939	(Celanese): Yarn is spun containing a morpholine derivative.
514,025	Oct 27, 1939	(Celanese): Pad calcium hydroxide, with or without benzylethylaniline.
518,908	Mar 11, 1940	(Celanese): Phenyl morpholine or p-phenylenedimorpholine.
527,834	Oct 17, 1940	(Kodak Ltd): Pad or codye melamine, N-acetyl, N-amyl, or N-phenylmelamine.
539,486	Sept 12, 1941	(Celanese): Pad triethanolamine with or without borax.
543,594	Mar 4, 1942	C H Giles, D G Wilkinson (Imperial Chemical Ind): 2-Methylbenzimidazole, 2-methylimidazoline, etc. See U S 2,340,375.
547,893	Sept 16, 1942	C H Giles, D G Wilkinson (Imperial Chemical Ind): N,N′-Diphenylformamidine and acetamidine, etc. See U S 2,369,122.
548,876	Oct 28, 1942	J M Tinker, D E Kvalnes (du Pont): Phenyl- or substituted phenylbiguanides.
558,784	Jan 20, 1944	B Collie, C H Giles, D G Wilkinson (Imperial Chemical Ind): N,N′-Diphenylethylenediamine, etc. See also U S 2,416,380.
563,181	Aug 2, 1944	(Celanese): Hydrolyze fabric with 23-40% caustic. See U S 2,366,241.
569,557	May 30, 1945	H C Olpin, S A Gibson (Celanese): Melamine-formaldehyde condensation products.
571,056	Aug 3, 1945	H Dreyfus (Celanese): Pad with aqueous triethanolamine containing a swelling agent such as ethanol. See U S 2,409,257.
571,971	Sept 17, 1945	(Celanese): 3-Amino-4-heptanol, etc. See U S 2,409,325.
607,085	Aug 25, 1948	H C Olpin, W B Miller, S A Gibson (Celanese): Yarn spun containing an ethylated melamine-formaldehyde resin.
612,601	Nov 15, 1948	(Celanese): Treat with aqueous-alcoholic hydroxyethylethylene diamine. See *J Textile Inst* **40**, A 373 (1949).
628,022		R J Mann, H C Olpin (Celanese): Process for application of an ethylated melamine-formaldehyde resin. *J Soc Dyers Colourists* **66**, 160 (1950).
636,501		W H Hindle (Celanese): Pad an alkali-metal hydroxide together with alcohol and thiocyanate swelling agents. *J Soc Dyers Colourists* **66**, 457 (1950). See also U S 2,495,810.
641,459	Sept 8, 1950	H C Olpin, W B Miller (Celanese): N-Phenylbenzamidine, etc. *J Soc Dyers Colourists* **66**, 595 (1950).

645,761	Nov 8, 1950	(Celanese): High acetyl yarn saponified to normal average acetyl. *Fibres 11*, 435 (1950).
646,832		H W Grimmel, A Guenther (General Aniline and Film): Naphthopyridine derivatives, *Brit Rayon & Silk J 28*, Dec, 1952, pp 63-4, 66.
647,220		R T Olsen (General Aniline and Film): Formaldehyde-amine-dicyandiamide resin. *J Soc Dyers Colourists 67*, 156 (1951).
648,631		H W Grimmel, J F Morgan (General Aniline and Film): N-Benzylaminotriazoles and tetrazoles. See also U S 2,529,-016.
648,651		H W Grimmel, A Guenther (General Aniline and Film): 1,3-Dibenzylimidazolidine, etc.
654,788		H W Grimmel, A Guenther (General Aniline and Film): 1,4-Dibenzyl piperazine, etc.

FRENCH PATENTS

694,750	Apr 29, 1930	Diisoamylthiourea; tetramethyldiaminodiphenylmethane.
877,139	Nov 27, 1942	N-Benzoylphenylenediamines.

GERMAN PATENT

740,983	Ring-substituted 3-benzoylaminobenzenes.

Meeting Report—Pacific Southwest Section

March 28, 1952
Nikabob Cafe, Los Angeles, Calif

THIRTY-THREE members and guests attended the Pacific Southwest Section dinner meeting at the Nikabob Cafe, Los Angeles, California, on Friday evening, March 28.

The results of the recent election of officers for the coming year were announced, and the new officers introduced, as follows:

Chairman—Norman Koehler
Vice Chairman—Robert Butler
Secretary—Angus Roberts
Treasurer—Fred Haselhorst
Councilor—Robert Mishell
Sectional Committee—John Smith, Robert Andersen, Theodore Smock, June Ericson

The program consisted of a showing of the movie, *Kon Tiki,* as the originally-scheduled speaker was unable to appear. The financial and membership reports presented by the treasurer and secretary respectively indicated that definite progress had been made in both areas during the past year.

Respectfully submitted,
JUNE ERICSON, *Secretary*

Membership Applications

(Concluded from Page P291)

Joseph S Warner — Dye Research Lab Tech, Celanese Corp, Cumberland, Md. Sponsors: W T Hagerty, E W Price.

Charles K Watson—Text Chem, North American Rayon Corp, Elizabethton, Tenn. Sponsors: A R Thompson, S L Hayes.

JUNIOR

Rupert H Austin, Jr — Dyer, Esquire Processing Co, Inc, Bondsville, Mass. Sponsors: W F Brosnan, F T Norris.

William S Barnard—Chemist, National Lead Co, Brooklyn, N Y. Sponsors: K L Barnard, L A Fluck.

Henry S Ebrhardt—Asst Chemist, Excelsior Finishing Plant, Pendleton, S C. Sponsors: E S Olson, J E Livak.

Stanley T Holland, Jr—Overseer, Dan River Mills, Inc, Danville, Va. Sponsors: J W Sutton, H M Chase.

Laurence A Kluger—Lab Tech, American Aniline Products, Hawthorne, N J. Sponsors: R W Brewer, H P Baumann.

John M Ross, Jr—Textile Chemist, The Borden Co, Chemical Div, Bainbridge, N Y. Sponsors: J M Myers, E E House.

William F Turner — Chemist, Research Dept, Franklin Process Co, Providence, R I. Sponsors: R W Joerger, W B Fish.

Daniel Young, Jr—Shift Overseer of Dyeing, Dan River Mills, Inc, Danville, Va. Sponsors: W E McMann, H A Chase.

ASSOCIATE

Armando Bachmann—Agent Distributor for Geigy Dyestuffs in Uruguay, Montevideo, Uruguay.

George A Emerson—Salesman, Procter & Gamble Distr Co, Cincinnati 1, O.

Laurence M Flamberg—Junior Chemist, National Aniline Div, Allied Chemical & Dye Corp, New York, N Y.

Harry Haskel—Research Director, National Coat & Suit Industry Recovery Board, New York 1, N Y.

Jose de Abreu Coelho—Lima Chemical Industrie Engineer, Fabrica de Tecidos de Linho e Algodao de Albano M Coeho Lima, Pervidem, Guimaraes, Portugal.

Alfred Lauchenauer — Textile Research Chemist, Raduner & Co, A G (textile finishers), Horn (thg) Switzerland.

Roger G Carr—Sales Trainee, Geigy Co, Inc, Chicago, Ill.

William M Meyer — c/o Amalgamated Chemicals & Dyestuffs Co, Ltd, "Neville House," Bombay, India.

Shirley A Walker — Textile Instructor, University of California, Los Angeles, Calif.

STUDENT

Samuel W Boddie — Clemson College. Sponsor: J Lindsay, Jr.

Louis G Darby—Clemson College. Sponsor: J Lindsay, Jr.

Gettys N Harris—Clemson College. Sponsor: J Lindsay, Jr.

Lonnie T Howard — Clemson College. Sponsor: J Lindsay, Jr.

George A Hutto, Jr—Clemson College. Sponsor: J Lindsay, Jr.

Charles B Simpson — Clemson College. Sponsor: J Lindsay, Jr.

Fitzhugh L Wickham—Clemson College. Sponsor: J Lindsay, Jr.

Wallace A Baker—New Bedford Textile Institute. Sponsor: F Tripp.

Robert E Griswold—New Bedford Textile Institute. Sponsor: F Tripp.

Betsy M Beil—New York State Institute of Applied Arts & Sciences. Sponsor: L R Henry.

Joseph L Davidman—New York State Institute of Applied Arts & Sciences. Sponsor: L R Henry.

Richard M Degan—New York State Institute of Applied Arts & Sciences. Sponsor: L R Henry.

David B DeMassa—New York State Institute of Applied Arts & Sciences. Sponsor: L R Henry.

Robert T Holland—New York State Institute of Applied Arts & Sciences. Sponsor: L R Henry.

Edward Janis—New York State Institute of Applied Arts & Sciences. Sponsor: L R Henry.

Herbert M Kagi—New York State Institute of Applied Arts & Sciences. Sponsor: L R Henry.

Herman Rosenberg—New York State Institute of Applied Arts & Sciences. Sponsor: L R Henry.

Gil M Dias, Jr—Bradford Durfee Technical Institute. Sponsor: J Watters.

William H Marsden — Bradford Durfee Technical Institute. Sponsor: J Watters.

Clyde M Garrison, Jr—North Carolina State College. Sponsor: H A Rutherford.

Richard A Gunthner—Philadelphia Textile Institute. Sponsor: W H Hughes.

APPLICATION FOR TRANSFER TO SENIOR MEMBERSHIP

David G Hedberg, Jr
Christian W Holweger, Jr
Samuel H Kassan
Alvin Lubin
Edward R Ravenel

• PATENT DIGEST •

PAUL WENGRAF

FINISHING NYLON OR SARAN——Durable Calendering Effects G, 4

U S Pat 2,577,957
(Aspinook Corp—Farnworth, Wood, Jr—Dec 11, 1951)

All kinds of resin precondensates have been recommended as finishes for cotton, rayon or wool, especially amino resins (i e, precondensates of the urea-, thiourea- or melamine-formaldehyde type either in alkylated form or mixed with sizing or filling agents).

The inventors have observed that fabrics woven from thermoplastic materials, particularly nylon or Saran, and impregnated with one of these condensates show considerably different effects after curing or calendering. It has been found that the resins *combine with the fibers, adhering permanently* to the thermoplastic material. It is essential that the impregnated goods be subjected for a short time in the calendering operation to a temperature approaching the melting point of the fiber so as to soften up the threads. This step provides sufficient penetration. The next process consists of curing the amino resin at a temperature substantially lower than that applied in calendering.

Embossing calendering effects also are within the scope of this invention. When engraved calender rolls are used, the pattern that remains after curing shows resistance to several laundering procedures.

Example: a urea-formaldehyde precondensate is mixed before padding with diammonium phosphate as a potential curing catalyst and with a softener of the fatty alcohol sulfate or fatty acid sulfonate class. Nylon cloth is run through this solution, first being damp-dried (to contain 5-10% moisture) on a tenter frame, then being passed in 0.5 seconds through the engraved rolls of an embossing calender at a temperature of 300°F, and finally being cured for 6 minutes at 190°F. The finish is durable to 5 soapings. Urea-formaldehyde condensates are used amounting to 3-10% of the fabric's weight while use of melamine resins requires lesser concentrations.

Among other references cited by the Patent Office:

USPat 2,481,809 (Polaroid Corporation—1949): a method whereby a thin, scratch-resistant layer of urea-formaldehyde or melamine-formaldehyde resin is formed on the surface of a thermoplastic substance (vinyl polymers, cellulose esters). This is especially useful in preparing optical lenses.

USPat 2,370,362 (American Cyanamid—1945): a cloth finishing composition comprising an emulsion of the oil-in-water type, the oily phase containing an alkylated melamine-formaldehyde plus an alkyd resin.

USPat 2,121,005 (Raduner, Bener—1938): calender finishes consisting of resin impregnation, drying to a slightly moist condition, embossing at about 150° C and curing at 110° C.

USPat 2,049,217 (Resines et Vernis Artificiels—1936): creaseproofing with a neutral solution of formaldehyde and ammonium sulfide or ammonium thiocyanate in the presence of urea, or, alternately, with solutions of thioaldehyde plus urea.

The inventors reportedly are aware that resin finishes resistant to calendering effects have been proposed in several earlier references. The present invention consists of applying these condensates to nylon or other thermoplastic goods, which obviously form characteristic, wash-resistant complexes with the resin condensates.

DYEING CELLULOSE ACETATE WITH ACID DYES—— Thiocyanate plus Glycols as Swelling Agents C, 4, 01

Brit P 661,800
(Sandoz Limited—Nov 28, 1951)

The dyeing of cellulose acetate fibers with dyes other than the water-insoluble dispersed acetate types has frequently been described. Swelling agents and/or solvents have been proposed for applying water soluble dyes including lower aliphatic acids, phenols, alcohols, thiocyanates, etc. The use of particularly large amounts of thiocyanates has been recommended whereby fixation can be effected by simple drying (see references below).

This specification contends that excess thiocyanate might damage the fibers, and furthermore, that acid azo dyes dyed or printed on acetate of cellulose might be impaired by reduction when applied in the presence of considerable amounts of thiocyanates. In contrast to other suggestions for printing or dyeing acid dyes on cellulose acetate, this one insists that the paste or padding solution contain several different swelling agents such as urea, thiourea or guanidine, a lower aliphatic carboxylic acid and an aliphatic hydroxylic compound, e g, glycol or glycolether, in addition to the thiocyanate, which is limited to 3% of the dye bath or printing paste. The fixation of the acid dye is reported to be brought about by simple drying at 40-50°C, washing first with cold water, then soaping at 60°C. The final dyeing or printing effect is stable to a treatment in a boiling, soap-containing bath. *Example:* a dyebath for an all-acetate fabric is composed of 7.5 g Acid Violet 4 BNS (CI #698), 100 g tragacanth thickener, 100 g ethylene glycol, 100 g thiourea, 30 g ammonium thiocyanate, 100 g acetic acid 100%, and the balance, water.

Other examples refer to other acid dyes such as Xylene Fast Green B, Azorhodine 2G, Brilliant Sulfon Red B, Xylene Blue AS, etc. A level dyeing effect on acetate-silk fabrics is obtained with Alizarine Light Green, according to the same method.

In this dyeing or printing method, no steaming aftertreatment whatsoever is required—a great advantage.

The preamble to this specification states that the use of swelling agents, and also of lower aliphatic agents and alcohol type solvents, is known per se in dyeing cellulose acetate with water soluble dyes. (Reference may be made to the various Celanese patents, e g, USPat 2,518,644, *Am Dyestuff Reptr 40,* 67, 1951; USPat 2,428,833—36, *ibid 37,* 397, 1948, etc). An excess of thiocyanate is recommended in Un Turkey Red Co's Brit P 588,106, *Am Dyestuff Reptr 37,* 243, 1948. The present invention points at the inconveniences in applying large amounts of thiocyanate. Possible drawbacks are avoided by substituting the major part of thiocyanate by alcohols, lower acids and compounds of the urea class.

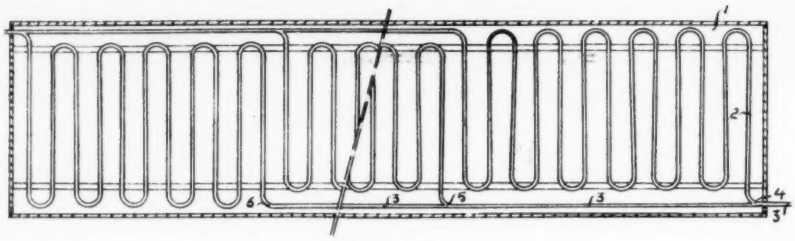
The parallel USPat 2,574,806 (Wiazmitinow and Goorhuis) was granted on Nov 13, 1951.

PRINTING DEVICE——SCREEN PRINTING TABLE——Concrete Top Heated by Zig-Zag Tubes D, 4

Brit P 662,181
(Naam Venn P F van Vlissingen—Dec 5, 1951)

Screen printing tables combined with drying chambers are in frequent use (cf, among others, USPat 2,452,329, *Am Dyestuff Reptr 38,* 477, 1949). Metallic-top printing tables heated by air convection are also known. According to the present specification, the latter type has to be considered as impracticable because the top is deformed by expansion.

The present patent proposes a top constructed of heavy material (especially concrete) in which heating tubes may be embedded. The accompanying drawing shows



Brit P 662,181

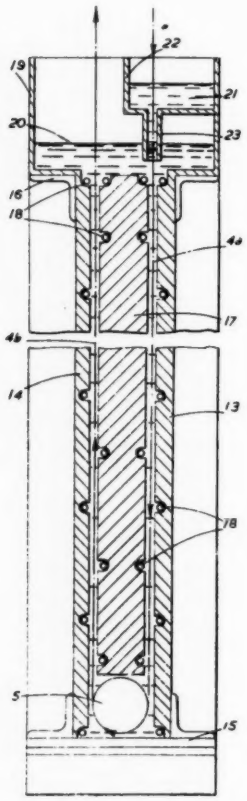
the tubes [2] in serpentine form. Warm water (instead of steam) is supplied by pipe [3], which is connected at points [4, 5, and 6] with tube [2].

Advantages are that the table temperature is kept constant and distortion of the table top is avoided.

DYEING VAT DYES—— Development in Molten Metal Bath, Antioxidants Added C, 2, 01

Brit P 663,066
(Standfast Dyers & Printers Limited—Hannay, Kilby—Dec 19, 1951)

French Pat 900,758 (I G Farbenindustrie, cf *Am Dyestuff Reptr 40,* 338, 1951) proposes quite generally that dyeings be developed by a passage of the goods through liquid metal alloys. This process was not only restricted to dyeing aftertreatments but to some other finishing operations, such as creaseproofing or waterproofing.



Brit P 663,066

Morton-Sundour Fabrics subsequently obtained Brit P 620,584 (1949), which refers especially to vat- or sulfur dyeings on the same principle. Continuous lengths of fabrics are first impregnated with a hydrosulfite-containing vat and then passed through the liquid metal alloy at temperatures between 60-100°C. The aqueous vat floats on the surface of the molten metal alloy, which is preferably composed of 13.3% tin, 26.7% lead, 50% bismuth and 10% cadmium. This patent also protects the apparatus used in this process: the surface of the metal [20] is indicated in the drawing; the dye liquid is contained in box [22]. Fabric [4a-4b] moves in the direction indicated by the arrows through the narrow channel in the alloy. (No drawing is shown in French P 900,758).

The present patent, assigned to the Standfast Dyers and Printers Limited, indicates an additional improvement over the main patent, BP 620,854. It has been observed that the hydrosulfite contained in the vat may attack the molten metal, thereby forming a sludge of sulfides on top of the alloy. This sludge frequently adheres to the goods and spoils the dyeing effect. To counteract this, an antioxidant should be added to the vat. This antioxidant must not appreciably retard the reducing action of the hydrosulfite, however. Suitable compounds recommended include reducing sugars (e g, glucose), benzaldehyde or tannic acid and related products. *Example:* a liter of the dyebath contains 150 g Caledon Jade Green XNS, 20 g NaOH, 30 g sodium hydrosulfite, and 15 g glucose syrup. Other examples propose that the glucose syrup be replaced by 20 g benzaldehyde or 10 g tannic acid, respectively.

PRINTING THICKENER—— Styrene——Maleic Anhydride ——Divinyl Benzene Terpolymer D, 1

Brit P 663,769
(Monsanto Chemical Co—Seymour—Dec 21, 1951)

This patent is parallel to USPat 2,533,635 (cf *Am Dyestuff Reptr 40,* 280, 1951).

FUMEPROOFING ACETATE DYEINGS (AMINO ANTHRAQUINONES)—— Triphenylguanidine C, 4, 07

Brit P 664,079
(Imp Chem Ind—Todd, et al—Jan 2, 1952)

It is well known that acetate dyes of the amino anthraquinone or substituted amino anthraquinone class are most sensitive to gas fumes. Dyestuffs particularly mentioned in this category are aminomethylanthraquinone (violet), tetraaminoanthraquinone (blue) and hydroxyethylaminodihydroxyanthraquinone (blue-green).

Many agents have been recommended to combat this sensitivity, for example, Brit P 340,572 recommends nitrogen bases, such as alkyl-, aryl- or aralkyl derivatives of urea, thiourea or guanidine. It is required that the products used remain efficient throughout the useful life of the textiles; moreover, the agents must not be volatile or removable by washing or dry cleaning. In certain cases, gas fading inhibitors turn yellow on exposure to light, unfavorably changing the original shade.

It has been observed that N-N′-N″—triphenylguanidine, a compound phenyl substituted on each N-atom, especially fulfills all the requirements enumerated above. It is taken up by the acetate fiber in a similar way and approximately at the same ratio as the dyestuff itself, it cannot be easily removed by washing, and it is not sensitive to light or gas fumes. The melting point is 145°C, thus it is not volatile or fusible at standard dyeing temperatures. In practice triphenylguanidine is dispersed with an Igepal-type compound, i e, an ethylene oxide condensate of octyl cresol, and pasted up with Carragheen mucilage.

Example: acetate fabrics are dyed with tetraaminoanthraquinone (Cellitone Blue), adding the dispersion of triphenylguanidine either to the dyebath or aftertreating the dyed goods in a fresh bath with this dispersion. Superior fastness to gas-fading may be noted.

Phenyl-substituted diamines have been recommended to serve the same purpose in USPat 2,416,380 (assigned to the same company, cf *Am Dyestuff Reptr 36,* 419, 1947). It appears that the inhibiting effect is greatly improved by applying triphenyl substituted guanidines.

STABILIZING CELLULOSE TEXTILES (Shrinkproofing)— Combined Glyoxal-Formaldehyde-Amine Condensate G, 2, 04

U S Pat 2,574,114
(Bozel Maletra——Lehmann, Lintner——Nov 6, 1951)

The preamble to this patent refers to former references regarding the stabiliza-

tion of cellulose with either formaldehyde or glyoxal. Drawbacks of formaldehyde treatments are 1) their volatility, which causes uneven results and 2) difficult working conditions. Glyoxal has the disadvantage of a considerably slower reacting time than formaldehyde, thus requiring higher reaction temperatures. Moreover glyoxal is found to be rather unstable in alkaline media.

The inventors suggest that urea, melamine or other resin-forming nitrogenous compounds be condensed first in an acid medium with glyoxal under conditions that allow only a portion of the active nitrogen (preferably less than 50%) to condense. The product is then reacted in an *alkaline medium* with up to 45% of formaldehyde, consequently resulting in a mixture of a glyoxal formaldehyde condensate with methylolurea, which is reported to be stable and nonvolatile with aqueous vapors, but nevertheless capable of giving the reactions of free formaldehyde without the risk of self-condensation to higher water-insoluble products. Stable, easily-reacting and odorless compounds that give a uniform effect are claimed. These precondensates are readily catalyzed by slightly (potentially) acid salts, such as ammonium nitrate.

Example: 1.6 kg urea dissolved in an aqueous solution containing 5.8 kg glyoxal and slightly acidified with HCl is boiled and cooled down. The precipitating condensate is separated and further condensed with an alkaline, glycerine-containing solution of formaldehyde. An aqueous solution of this condensate, containing about 60 g of dry substance per liter and about 20 g of ammonium nitrate, is used for impregnating the goods. Further procedure consists of squeezing to a pick-up of 100%, drying at 80-90° C and curing at 120° C. Swelling of the cellulose fiber in this case was reduced from an initial value of 100 down to 53; the shrinkage was 0.5% compared with the original figure of 15%. It may be assumed that the polyalcohol (glycerol) adds to the stabilizing effect of methylolurea. In another example glycerol was omitted without impairing the final results.

Among other references cited by the Patent Office:

U S Pat 2,412,832 (Cluett Peabody/1946): stabilizing cellulose by impregnating with aqueous glyoxal, an acid catalyst and an amino resin precondensate and then baking (cf *Am Dyestuff Reptr 36,* 337 (1947)).

U S Pat 2,388,086 (Montclair Research/1945): ketone and glyoxal are reacted under either acid or alkaline conditions for preparing resins, which may be used for creaseproofing or durable finishing purposes.

U S Pat 2,301,676 (General Aniline/1942): water repellent agents consisting of quaternary ammonium (pyridinium) salts condensed with ester-like reaction products of chloroacetic acid and halogen methyl derivatives of higher alcohols.

Brit P 510,199 (Courtaulds/1939): reduction of the swelling of cellulose by impregnating with at least 6% of formaldehyde, acid catalyst and not more than 2% of urea or thiourea, and curing at 100-150° C.

Brit P 458,877 (I G Farben/1936): creaseproofing goods by impregnating with melamine-formaldehyde solutions and heating to elevated temperatures (145-150° C).

Reference is further made to the shrinkproofing process covered by U S Pat 2,562,161 (Cluett Peabody, cf *Am Dyestuff Reptr 40,* 778 (1951)), which suggests the use of acid amides condensed with glyoxal. Further references are mentioned in the digest of U S Pat 2,412,832 (*Am Dyestuff Reptr 36,* 337 (1947)).

VAT DYEING WITH DYES, SENSITIVE TO OVER REDUCTION AND PYRANTHRONE DYES——Nitrogen Compounds Added C, 4, 02

U S Pat 2,576,846-47
(American Cyanamid——Mecco——
Nov 27, 1951)

These two specifications should be studied in connection with the same inventor's U S Patents 2,548,544-45, which are digested in *Am Dyestuff Reptr 40,* 538 and 605 (1951), respectively.

U S Pat 2,576,846 refers to the dyestuff brands enumerated in U S Pat 2,548,544. While the latter patent suggests that over-reduction of these high-temperature-sensitive vats be avoided by adding alkali halogenates (preferably sodium chlorate) to the dye bath or the printing paste, the newer patent recommends the use of compounds in which an N-atom is linked by at least one valence to an element other than O or N. Numerous examples of these types of compounds are offered, e g, nitroparaffins (particularly nitropropane), and aromatic nitro compounds, such as nitrobenzene, nitroso compounds, e:c. Another mentioned compound is hydroxylamine potassium disulfonate, $HON(SO_3K)_2$, which is prepared from potassium nitrite and potassium acetate by passing SO_2-gas at low temperatures. (See *J Am Chem Soc 54,* 977 (1932))

These nitrogen compounds are added to the dyestuff at a minimum ratio of 1:4. Larger amounts of the nitrogen compound are not critical, but a ratio exceeding 1.5-2 parts nitrogen compound to 1 part dye does not appreciably improve the results. The effect may not be due only to buffering the reduction because other oxidizing agents, such as inorganic nitrates, organic nitrites or nitrates, will not give the improved results obtained by the present method.

Example: cotton yarn is dyed for 60 minutes in a vat containing Indanthrene Blue GCD or Ponsol Blue BF (C I 1113), NaOH, hydrosulfite and nitropropane at a temperature of 200° F, after which it is reoxidized in an acetic acid-sodium peroxide solution. The shade obtained is a brilliant blue, as contrasted with a dirty, dull gray obtained by control dyeing wherein nitropropane is omitted.

U S Pat 2,576,847 recommends the use of the same nitrogen products in dyeing the golden orange vat dyes Pyranthrone (C I 1096), and dibromopyranthrone (C I 1098), which otherwise show a loss of strength and brilliance at elevated temperatures. In contrast to the dyestuffs indicated in the co-pending U S Pat 2,576,846, the effect cannot be due to a reduction-buffering reaction because the Pyranthrone dyes are not decomposed or over-reduced in boiling dyebaths. Thus the general explanation given for dyestuffs that are over-sensitive to over-reduction cannot be tenable for Pyranthrone and its halogenated derivatives. Nevertheless, considerably improved results in dyeing at higher temperatures can be noted although the factors causing this effect are not known.

Example: cotton yarn is dyed at 200° F for 60 minutes, in a vat, the bath consisting of 0.09 p of the dye C I 1098, 3 p NaOH, 3 p sodium hydrosulfite and 1.5 p nitropropane in 400 p water. After reoxidizing (as above) and soaping as usual, a bright orange is obtained; a dull and weak yellow shade is obtained under analogous conditions when nitropropane is left out.

References cited by the Patent Office (for both patents):

U S Pat 2,029,999 (Ciba/1936): fixing various dyes (vats, among others) by using aromatic or aliphatic sulfonamides, e g, dibenzene sulfonamide, as auxiliary agents.

U S Pat 2,146,646 (General Aniline Works/1939): adding sulfobetaines to vat printing pastes.

U S Pat 2,383,393 (American Cyanamid/1945): substituted quinone sulfonamides, e g, beta anthraquinone sulfonic acid, reacted with acetamide and used as an intensifier for vat printing pastes.

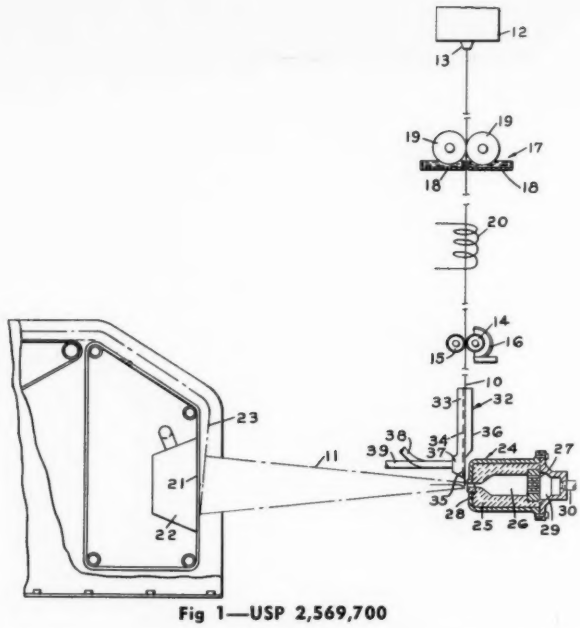


Fig 1—USP 2,569,700

COLORING GLASS FIBERS—— Covering with a Fused Colored Glass Film C, 4, 07

U S Pat 2,569,700
(Owens Corning Fiberglas Corp——Stalego——Oct 2, 1951)

This specification discusses past attempts at coloring glass fibers. Difficulties have been experienced in the main because of the poor dyestuff adsorption of this material.

The problem is to obtain fine glass threads in which the color is durable, fast to crocking, and of sufficient intensity. This problem is attacked by covering the fiber surface with a coloring substance in a fused state having approximately the same melting point as the glass material that is transformed into fibers. The coloring substance contains mostly metal oxides blended with glass particles of the same composition as the uncolored glass threads to be covered by the fused mixture. The blend of metal oxide and glass particles is melted to produce a homogenous compound, then cooled to room temperature and reduced to a fine powder. Sodium- or lithium silicates may be added as bonding agents. This powder is pasted with water to the necessary consistency for applying on the melted glass in the spinning process.

Fig 1 illustrates this new method. Glass batch is fed to the feeder [12], containing several orifices [13] through which melted glass filaments flow downward through rolls [19]. A pasty mass of the coloring-pigment-and-glass composition is supplied from tank [18] and the layer formed is fused onto the filaments by a heating device [20]. Glass filaments covered with this colored layer are sufficiently cooled down before arriving at rollers [14]. Here they are gripped and guided through [32, 33] to the orifice of a combustion chamber [26], heated again to relatively high temperatures (2500-3000° F) and hurled at a great velocity by means of expanding gas against a conveyor [21], which transports the comminuted fiber mass in form of a mat. A suction chamber [22] is placed on the other side of the (foraminous) conveyor to assemble the fibers, which are ultimately deposited on [21].

Among other references cited by the Patent Office:

U S Pat 2,457,777 (Owens Corning Fiberglas/1948): an apparatus for guiding and threading glass fibers, comprising a removable guiding portion with a number of grooves corresponding in number to the lateral spacing of the guide channels.

U S Pat 2,450,363 (Owens Corning Fiberglas/1948): producing very fine glass fibers by feeding glass rods or filaments into a high speed and temperature-gaseous blast and projecting the material on a conveyor, where a fine mat is formed.

U S Pat 2,433,116 (Westinghouse Electric/1947): manufacturing glass tubing, the inner side of which is covered by electrostatic precipitation with a fluorescent powder.

Brit P 381,582 (Rössler/1932): producing glass threads by melting the material in a furnace. The threads pass from the furnace, through an opening so narrow that only the very fluid material can enter, and then to a cone.

Further reference is made to several other patents assigned to the same Company. The formation of a surface layer containing dyestuffs, a plasticized glue and resin precondensates is described in U S Pat 2,428,302 (cf *Am Dyestuff Reptr* 37, 383 (1948)). Coating with aluminum silicates, which act as mordants for various dyes, or with organosilicon compounds as mordants for basic dyes is recommended in U S Pats 2,433,292 and 2,436,304, respectively (*Am Dyestuff Reptr* 37, 568 (1948)). The present method provides for solidly-colored surface layers, though it is restricted to inorganic melt-resistant pigments.

SHRINKPROOFING WOOL—— Divinylsulfone Treatment of Proteins in General A, 2, 04

U S Pat 2,579,871
(U S Rubber Co——Schoene——Dec 25, 1941)

This patent generally refers to a modification of all kinds of proteinaceous substances by reacting them with divinyl sulfone $(CH_2{:}CH)_2SO_2$. Applications of this reaction, as mentioned in the examples, consist of preparing horn-like masses or gels from gelatin, casein, soybean protein or zein, further tanning hides, etc. In the field of woolen textile processing shrinkproofing effects and stabilization against alkalis might be noted particularly (examples 8 and 9).

Wool (and proteins in general) are treated with divinylsulfone, preferably in the presence of a small amount of an alkaline catalyst, but in some instances alkali might be omitted entirely. The protein is moistened with an aqueous dilute solution of divinylsulfone and after-treated with dilute alkali. The effect of this reaction, which proceeds at room temperature, is conspicuous in processing hides that become resistant to boiling water in contrast to untreated skins that shrink rapidly and partially disintegrate in hot water at 50-100° C. But surprising effects in wool treatments can also be observed.

Alkali hydroxides, alkali phosphates, quaternary ammonium hydroxides or tertiary amines can be used as catalysts.

Example: a piece of wool flannel was dipped in a solution of 3% divinylsulfone and 2% NaOH, squeezed to a 200% pick up and kept in that state for an hour at room temperature, after which it was washed in dilute acetic acid, rinsed and dried. The shrinkage was about 15% compared with 30% shrinkage of a parallel sample in which divinylsulfone was omitted. This wool treated in a 5% alkali hydroxide solution was found to be substantially unaltered in contrast to normal wool and alkali-pretreated wool, which completely disintegrated under the same

conditions.

Among other references cited by the Patent Office:

U S Pat 2,290,294 (J A Benckiser/1942): superior tanning effects obtained by using reaction products of anhydrous phosphoric acid (87% P_2O_5) and phenol formaldehyde.

Brit P 471,010 (Geigy): use of aromatic sulfones as tanning agents, e g, dioxy diphenylsulfone, dioxy dimethyl diphenylsulfone; no aliphatic unsaturated sulfones are mentioned. *J Org Chem 11,* 719-735 (1946) describes the reactions of divinylsulfone with water, thiosulfate, thiophenol, sulfhydryl compounds (in general), pyridine and other bases.

U S Pat 2,524,399 (cf *Am Dyestuff Reptr 40,* 527 (1951), Geigy—Schoene and Chambers), refers to an analogous treatment for stabilizing (shrinkproofing) cellulose, preferably rayon, with divinylsulfone.

INDIGOSOL DYEINGS on Two-Sided Fabrics——One Side Development by Light Exposure C, 4, 03

U S Pat 2,580,873
(Dan River Mills——Womble——Jan 1, 1952)

This invention is based on the property of some Indigosol dyes or analogous products ("Algosols", "Solvats" and some "Ponsol Soluble" dyes) to be developed, i e, oxidized, by strong light exposure, preferably by carbon arc light. The sulfuric acid esters of leuco vat dyes, generally known as "Indigosols", have little affinity for most textile materials, especially vegetable fibers. They can easily be removed in the unoxidized state by washing. By application of strong sunlight or artificial light of the proper wave length and intensity they are developed and insolubilized on the fiber.

This property, common to many members of this dyestuff group, is useful in applying the dyestuff solution to the uncolored fabrics and *thereupon exposing one side only* to actinic light rays. After the fabric is soured in a soap solution, the dye is removed from the unexposed side. A second step may follow——impregnating the fabric with an Indigosol dye of a contrasting shade and then exposing the other side to the light source. Yet another modification consists of impregnating the fabric with a mixture of an Indigosol (e g, blue) plus a pigment dye emulsion (e g, yellow or the like), removing the unfixed Indigosol and curing. In this event one side will show a green, the other, a yellow shade.

Example: mercerized cotton poplin is padded in a 2% solution of Algosol Pink I R extra (prototype 109). The wet fabric placed down flat on a smooth surface and exposed to light until the full shade appears. After scouring, the fabric is impregnated with Algosol Blue 04B (C I 1184); the uncolored side is exposed the same way to light and scoured again. A cotton fabric results that is dyed on one side with a fast bright red and on the other with a blue shade.

References cited by the Patent Office:

USPat 2,214,365 (Waldrich Co / 1940): printing fabrics by photographic means, i e, impregnating with a water soluble dye of the Indigosol class containing formic acid and exposing to light under a negative.

USPat 1,599,910 (Two-Tone Corporation / 1926): permeable fabrics having different shades on either side. These are obtained by spraying one side with one color and then the other with a color of contrasting shade.

Brit P (Haendel / 1930): printing light-sensitive diazosulfonates on a fabric without using a negative by printing mixtures of the components and exposing the fabrics to a strong electric light source.

WATER REPELLENT SYNTHETIC FABRICS—— Long Chain Substituted Phenol-Aldehydes G, 2, 02

U S Pat 2,582,239
(Secretary of War——Dodd——Jan 15, 1952)

The introduction to this patent states that greater difficulties have been experienced in rendering nylon or Vinyon ("noncellulosic synthetic textiles") water-repellent while more satisfactory results have been achieved hitherto by analogous treatments of cotton or other cellulosic materials. The contact angle of synthetic textiles, as the most significant criterion for water repellent effects, compares unfavorably with that of cellulosic fabrics. This phenomenon may be explained partly by the fact that the larger specific surface area of the cellulose provides a more extensive coating of the individual threads. A satisfactory water-repellent resin finish requires the application of highly-water-repellent, nonbrittle and sufficiently polymerized (sparingly soluble) condensates as well as a great mechanical strength of the cured resins, equalling at least that of the untreated original fibrous substance.

These requirements are said to be fulfilled by the application of formaldehyde condensates of long-chain-substituted phenol. The chain may consist of an alkyl or acyl of at least 12 C- atoms. A suitable example of this type of alkylphenol is the oil extracted from cashew nut shells (Cardanol) in an hydrogenated state. The original unsaturated compound, m- $C_{14}H_{27}C_6H_4OH$, is thereby transformed into m-$C_{14}H_{29}C_6H_4OH$ (meta-tetradecyl phenol), forming the necessary long-chain-bearing condensate. The corresponding o- and p-phenols are found to form a three-dimensional condensate of phenolic nuclei, linked by methylene groups; they proved to be less appropriate in this process than the m-compounds. In contrast, the o- and p- acyl substituted phenols, such as stearoylphenols, $C_{17}H_{35}CO.C_6H_4OH$, give the best results. Formaldehyde is applied in the form of hexamethylene tetramine or paraformaldehyde. The condensates are used in slightly alkaline emulsions.

An example calls for dissolving m-tetradecylphenol, and hexamethylene tetramine in isopropanol and refluxing the mixture until preliminary condensation takes place, removing the solvent, taking up the residue with a "water-nonmiscible" solvent and emulsifying this solution with ammonium stearate-water solution. A nylon fabric impregnated with ths emulson, made slightly alkaline by ammonia, is air dried and cured for several days in an oven at about 70° C. The fabric is made flexible and even stronger than the unprepared material. The water repellent effect persists after repeated laundering.

Because of the low curing temperature needed for polymerizing these resins, this method seems to be particularly adapted for heat-sensitive thermoplastic fibers, such as nylon or Vinyon.

Among other references cited by the Patent Office:

USPat 2,420,157 (American Cynamid / 1947: finishing textiles with an aqueous solution of a quaternary ammonium salt of a melamine-formaldehyde resin condensate and then heating to decompose the ammonium salt. This procedure is recommended for obtaining creaseproof and water repellent effects (cf *Am Dyestuff Reptr 36,* 578 (1947).

USPat 2,370,362 (American Cyanamid / 1944): high alkyl substituted phenols, e g, cashew nut shell liquid, used as a solvent for Rotenone (insecticide).

USPat 2,284,369 (Harvel Corp / 1942: hydrogenated Cardanol (a high alkyl-substituted phenol) is recommended for various technical uses, especially for forming resin condensates with formaldehyde.

USPat 2,220,856 (Heberlein Patent Corp / 1940): producing waterproofing effects by impregnating with organic solvent solutions of thiocyano groups containing ethers, e g, thiocyanomethyl octadecylether, and subsequently baking.

USPat 2,175,393 (Henkel & Cie / 1939): obtaining resinous products from phenols, olefins and aldehydes. Condensates of alkylphenols and aldehydes, catalyzed by perchloric acid, are used as film-forming and softening agents.

USPat 1,987,549 (Bakelite Corp / 1935): preparing paper with an oil soluble varnish from phenol-aldehyde condensate modified with an oil, e g, tung oil. Penetration is improved by emulsifying this composition with water plus gelatin, which emulsion breaks upon application on the substratum.

ABSTRACTS

The Future of Wool

M Lipson, *Textile J Australia* 26, 922-3, Nov 20, 1951.

The increased production of synthetics in the United States has caused considerable speculation about the replacement of wool by artificial fibers. Depending on individual interests, there are two major schools of thought, one predicting a catastrophe for the wool industry, and the other anticipating little if any effect. That two authoritative groups could reach such diametrically opposed conclusions indicates that the question, "What is the future of wool?", is a difficult one to answer. Both scientific and economic aspects come into the question, and each requires consideration before one can arrive at conclusions. Let us consider first of all some of the scientific data on wool and see how it compares with other fibers.

The wool fiber consists of two main types of cells making up the cortex or internal portions surrounded by the external layer of scales. Microscopic examination of the cortex, which forms the major part of the fiber, shows that it consists of long spindle-shaped cortical cells, which are joined by an intercellular material. Finally, by chemical analysis, we find that wool is a protein consisting of 18 different amino acids.

The scales cause a directional frictional effect in wool fibers which permits them to move in one direction only when washed or milled together. This is the basis of the milling process used in finishing, and is also the fundamental operation in the felt industry. No artificial fiber has been prepared possessing the felting properties of wool. It will be very difficult to devise artificials that have this feature.

The warmth of fabrics made from wool is largely due to the crimped nature of the fiber, which allows room for plenty of insulating air in the final fabric. The crimp in wool is permanent, whereas that imparted to many of the artificial fibers tends to disappear during use. It remains to be seen whether the latest American synthetics possess a crimp that is permanent.

An outstanding property of wool is its high resilience, or ability to recover from compression. In this respect, wool is better than any of the other fibers, with nylon a close second.

As regards moisture regain, wool has the highest value (15 per cent), while nylon has only 4.5 per cent and Orlon only 0.9 per cent.

The true synthetics are all much stronger than wool. Breaking strengths, however, do not give the full story as to how a fiber will function in practice, as conditions of wear may not subject each fiber to its maximum breaking load. A useful test is resistance to repeated bending, and wool performs well under these conditions.

The present increase in world population of 20 million per year, together with rising standards of living throughout the world, will, in the opinion of the author, require all the textile materials that can be produced, both natural and synthetic. With price stability, he sees no reason why all the wool that can be produced will not be consumed. It will probably be used, for many purposes, in blends with true synthetic fibers, and increased production of the latter can be expected. As a result of such blending it is likely that improved fabrics will result from wool.

It can be seen, therefore, that the advent of the new synthetic fibers should not result in a catastrophe for the wool industry, but that each type of fiber, improved through research, will play its part in helping to overcome the world shortage of textile raw materials.—WHC

Flame Resistance of Nylon Yarns

(from Du Pont Technical Service Bulletin)

Textile Age 10, 70-1, March, 1952.

Experience has shown that nylon yarn is more resistant to burning than cotton, rayon, silk, or wool. The natural flammability of clean, undyed, finish-free nylon and of nylon dyed and finished with properly selected materials is very low and considered highly satisfactory for practically all textile purposes. Nylon will not flash burn, but will melt at a temperature of approximately 480° F if a flame is applied. However, as soon as the igniting flame is removed, the melt will drop away from the yarn or fabric and harden with very little tendency for further flame propagation. The term "flameproof" as applied to fabrics implies a resistance to any spread of flame after the fabric has been ignited. Undyed, unfinished nylon yarn or fabric may, therefore, be considered flameproof, since it does not support spreading of the flame after the igniting source has been removed. Cotton, paper, and wood are distinctly different in this respect.

If pigments, dyes, oils, finishes, salts, or other materials are present on the fiber, there is always the possibility that the resulting treated yarn or fabric may support combustion due to their presence. Some of these materials may be flammable themselves, or may release oxygen on ignition, thereby speeding up the burning of nylon or other fibers on which they are present. Other materials, which are not flammable, such as antimony oxide, talc, etc, in powder form on nylon may increase its tendency to burn. Apparently, the molten drops of nylon are thickened by the powder, and instead of dropping off before the flame is propagated to adjacent fabric, the drops cling to the fabric due to increased viscosity. Additional material is then melted and ignited, thus propagating the flame. This explains why certain flame-retardants for other materials may increase the flammability of nylon.

Combination nylon-Fiberglas fabrics may burn when ignited, although neither nylon fabric nor Fiberglas fabric will support combustion alone.

Some tests were made in the laboratory to determine the effect of various types of dyestuffs on the flammability of nylon fabrics. In general, it was found that most of the acetate, acid, and direct dyes had only slight effect or none at all on flammability, but many of the chrome and other metallized dyes appreciably increased it.

In view of the many variables affecting flammability, it is considered advisable that each manufacturer of products made from nylon have flammability tests conducted on his own products as they are ready for the market, wherever flammability is an important factor.—WHC

Fluorescent Fault Detection: A Saver of Waste in Textile Printing

Anon, *Indian Textile J* 62, 170-1, December, 1951.

Continuous roller pattern-printing on textiles by modern methods, using naphthol dyes, discharge pastes, etc, includes speed of production among its advantages. This very speed, however, can involve high costs in spoiled materials unless faults in printing are seen and stopped as they occur. Since the dyes themselves are frequently invisible when printed, such a check is difficult to effect.

Several of the dyes used, however, possess the property of fluorescence; when exposed to ultraviolet rays, they show luminescent colors, sometimes of great brilliance. The Azoic, Naphthol, and

Rapidogen series are fluorescent as manufactured; the other dyes may be made to fluoresce by simple additions. Consequently, if the fabric is exposed to ultraviolet radiation immediately after printing, faults on the pattern—"doctor blade" streaks, roller streaks, etc—will show by their fluorescence, probably more brilliantly than the pattern itself, so that defective printing can be stopped immediately and spoilage of lengths of cloth thus prevented.

Vat dyes do not normally fluoresce; however, as mineral oil is often added in the color box, and since it possesses its own bright blue fluorescence, this will show in the print. Paste discharges are not naturally fluorescent, but many of the bleaching additives (Blankophor, Tinopal, Uvitex, etc) give sufficient fluorescence against the dark fabric to enable a check to be made, under suitable conditions.

The Hanovia "Textilite", an ultraviolet lamp designed expressly for the textile industry, is described. It consists of a low-pressure mercury vapor discharge tube 40" long mounted in an aluminum hood. The front of the hood is covered by a dark filter glass, which permits only ultraviolet radiation to pass. Suitably mounted, the lamp fluoresces the whole width of a 36-inch piece, and is so screened that it can be used while the normal intense printing lighting is also in operation. The author states that the lamp has found extensive adoption among English textile printers.—WHC

Synthetics Go to War. A Report of the Past, Present, and Future Uses of Man-made Fibers by the Military

D Koch, *Textile Forum* 9, 15, 46, February, 1952.

When World War II began, the United States found themselves somewhat short in their great need for natural fibers. Silk shipments ceased almost immediately, and the Japanese threatened the Australian wool source. The first use of synthetics was to replace the natural fibers of which supply was limited.

The first large demand for any synthetic fiber by the military was for nylon in the making of parachutes. Its importance cannot be underestimated because few articles require the consumer to be so completely satisfied as does the parachute. Soon rayon found its way into that field after research had produced a medium tenacity yarn.

The tire program during the war, which called for upwards of 300,000,000 pounds annual capacity of high-tenacity viscose rayon, was by far the largest single use of a synthetic fiber.

Nylon, with its high tensile strength and its ability to give before breaking, has been used extensively for glider tow line. Viscose rayon, besides being used in tires, was used in self-sealing gasoline tanks for planes, the cord in transmission V-belts, and mixed with cotton yarn for uniforms.

Fiberglas has also been used by the military. Its fireproof quality makes it ideal for flare chutes and for curtains on combat ships.

Important requirements for military fabrics have been lightness of weight and resistance to moisture and fungus. Since the soldier carries his own tent or sleeping bag, lightness is essential. Nylon has met the requirements of these articles, as it is light and resists fungus. It has also proved its use for various types of screens and nets to keep out flying pests. In screens for tent openings that require flexibility for rolling without creasing, together with mildew resistance and waterproofness, millions of yards of Saran are being used.

It was found that spun nylon duck was many times more durable than duck made from cotton in resistance to external and internal abrasion. Experiments employing spun nylon in jungle boots indicated that its durability was equal to that of leather, while at the same time its porosity was controllable according to the designer's wishes.

Among the unique uses of synthetics during the war was the development of light weight, fragment-resistant armor made from laminated cloth known as Doron. Here, for the first time, the tensile strength of these filaments was used to stop high-explosive fragments in place of the compressive or shear strength of metal armor. Two types of this body armor were developed. One was a rigid type utilizing piles of high strength unidirectional glass fabric bonded with a thermosetting resin, and the other was made from high tenacity nylon fabrics which were made into thin pads of unbonded fabric.

When wool conservation was requested by the Munitions Board, substitution in woolen and worsted fabrics was made by the use of Orlon acrylic fiber and Dacron polyester fiber. Both of these fibers served adequately.—WHC

Determining Resins, Embedded in Creaseproofed Textiles

Graeser, *Textil-Praxis*, 823-24, November, 1951.

Creaseproofing effects are generally measured by determining the creasing angle. It may be necessary however to find out about the distribution of the resin within the treated fabrics. It is known that some acid dyes, such as Kitone Pure Blue V (Ciba) or Benzyl Red, color the resins, leaving the rayon fibers themselves uncolored.

Strips of the fabric extending over the entire width are dyed at 80° C for 20 minutes at a dyebath ratio of 1 to 20 with 0.5% of Kitone Pure Blue V. Dyeing an untreated fabric as a blank in the same bath is advisable in order to observe the difference. Both specimens are then rinsed until the blank again appears colorless. Melamine-resin finished goods have to be dyed at a pH less than 1 (HCl); other resin-finished strips may be dyed either at pH 8.8-9.0 or at pH 2.4-2.6, but acid dyeing is preferred in any case.

Shaded color effects point to irregular migration during drying, the depth of shade depending upon the resin concentration. The same criterion also applies to the degree of condensation—dark shades corresponding to an increased state of resin formation provided the test is carried out in acid baths. (Alkaline dyeing has the reverse effect, giving darker shades for poorly-condensed compounds).

One drawback of this test is that clear results are obtainable only on undyed fabrics. Extension of the method to dyed goods is contemplated. Unlevel creaseproof effects may be caused by irregular drying and migration or by inadequate curing temperatures. Complicated chemical and mechanical tests up to now are known only to determine the changing resin concentrations in finished fabrics. (In German)—PW

Dyeing Mixed Wool-Perlon Fabrics with "Lanaperl" Dyestuffs

Schleicher, *Textilber* 32, 779-80, October, 1951.

Perlon, a linear superpolyamide of the nylon group, has acquired increasing importance in European countries since World War II. Straight filaments or cut (staple) fibers are in use in the knitting industry and admixtures with other fibers (preferably wool) have become very popular in the manufacture of piece goods.

Dyeing wool-Perlon mixtures with dispersed acetate dyes is not advisable because the wool portion lacks fastness to crocking, to moisture and to light. Acid dyes can be applied well on both fibers but the limited affinity of polyamide fibers (5-10% that of wool) has to be taken into consideration. The amido groups do not take up dyestuffs in excess of this saturation value. Another characteristic dyeing property of the polyamide fibers is their selectivity. It is not gener-

ally known that monosulfonic acid dyes are predominantly absorbed while dyes bearing two or three sulfogroups are only taken up when free amidogroups are available. It is even possible for some dyes, e g, disulfonic acid dyes, already absorbed by the fiber to be driven out by an excess of monosulfonic acid dyes. Consequently, selected dyestuff groups have to be employed. Farbwerke Hoechst has developed such a series, the "Lanaperl" dyes, especially for uniformly dyeing wool-Perlon mixtures. The brands available to date are enumerated and formulas for application are offered. (In German)—PW

Acid Dyeings on Natural and Artificial Protein Fibers and Polyamides

Weber, *Textilber* 32, 865-868, October, 1951.

The author discusses the theory of acid dyeing in general, explaining the reactions that occur in this process. They can briefly be characterized by an addition of H- ions to the NH_2- groups of the wool molecule under strongly acid conditions, which increases the electropositive charge of the fiber, all consequently precipitating the electronegative dyestuff ions.

Watkins, Millson and Royer (*Am Dyestuff Reptr 36,* 45, 122, 1947) proved that absorption on the fiber's surface occurs almost instantly, followed by migration, which is at a maximum at high temperatures and pH values between 1.5 and 2.5 in routine wool dyeing procedure. This migration is modified not only by the nature of the epithelion layers surrounding the fiber's core, but also by the constitution of the dyestuff molecule. A striking practical example here is the difference in absorption speed of acid azo dyes (of yellow and red shades) in blends with (blue) acid anthraquinone dyes. Nonleveling dyes call for changed conditions, such as decreasing dissociation of sulfuric acid, by adding Glauber's salt, starting at low temperatures or replacing sulfuric acid by ammonium salts of organic acids.

Natural silk is generally dyed from soap degumming baths which contain sericin emulsified in the liquid. Sericin primarily binds the dye and releases it gradually during the dyeing procedure, thus acting as a leveling agent.

The relatively small affinity of nylon to acid dyes is explained by the absence of side-chain amino groups. The only amino groups available for reaction are located at the ends of the molecules. Eloed and Froelich demonstrated that theoretically nylon could be dyed with acid dyes in increased depth of shade under extremely acid conditions. It can be assumed in this case that peptide-type NH-groups acquire an additional charge; neither fastness to water nor tensile strength are satisfactory in this event, however. (In German)—PW

Molten Metal Baths in Textile Processing

Nestelberger, *Textilber,* 955-58, December, 1951.

The author discusses the story of molten-metal treatment of textiles. The drying of paper webs either by using liquid metal compositions or water immiscible hot oils was recommended as early as 1903. Consequently the scope of a German application of I G Farben, based on the author's experiments and corresponding to French Pat 900,758 (cf *Am Dyestuff Reptr 40,* 338, 1951), had to be restricted to "chemical treatments of *textiles* using molten metals". (Note: French Pat number 900,785, quoted in the original article appears to be a misprint).

The author first intended to develop a metallic coat on linen or cotton for protection in tropical countries. Resin compositions proved to be impractical for this purpose; thus the fabrics were run through melted aluminum at temperatures of 700-720° C. A device designed for this operation led to the first experiments in fixing vats analogous to the development in the "Colloresin ager" and the "Electrofixierer". Further work on Indigosol prints and discharges gave promising results. While in the author's laboratory experiments the impregnating bath was set apart from the molten metal container, the Standfast Dyers and Printers Ltd made progress in placing the dyebath immediately above the melted alloy, thus avoiding squeezing of the fabric after padding. Some problems still remain to be solved, e g, some vats are not satisfactorily fixed under these conditions. In an attempt to overcome some of these disadvantages a passage through air-free steam was inserted after the metal passage. Further experiments comprised pigment pad dyeing, direct dyeing and use of insoluble and soluble (Astrazone) dyes for acetate. (Results were described as "fair".) The same is said to be true for fixing pigments with resin precondensate emulsions: curing was carried out at 300° C within one second passage and the effect was equal to that of a 3 minute passage through a standard curing chamber heated to 140° C. Singeing and drying might also be possible with adapted equipment. An alloy of Sn- Bi- Cd was generally used when temperatures between 90-120° C were required. Lead had the drawback of producing rapid surface oxidation. Alloys for use at temperatures over 160° C were composed of Sn and Cd. Drawings of the various devices used in these experiments and other important details are presented. (In German)—PW

Developments in New Synthetic Fibers

H C Mersereau, *Canadian Textile J* 69, 57-9, March 28, 1952.

The author submits the following system by which most synthetic fibers can be segregated by type:

I. *Manufactured or Reprocessed Fibers:*
   (a) Cellulose based — viscose, acetate or cuprammonium rayons.
   (b) Protein based—Ardil, Vicara, Lanital, Aralac, etc.

II. *True Synthetic Fibers:*
   (a) Polyamides—nylon, Perlon L, Amilan, Enkalon, Grilon, Austrylon, etc.
   (b) Polyester fibers—Dacron and Terylene.
   (c) Ethylene based fibers—(deriving mostly from polyacrylonitrile and/or vinyl chloride) Orlon, dynel, Vinyon, Rhovyl, Acrilan, etc.

He admits that this classification does not suit all requirements. There is, for example, no place for the polyurethane fibers (Perlon U), protein-type fibers built up from amino-acids are not provided for, nor is there any provision for the growing class of modified natural fibers, such as the recently announced treatment of wool with beta-propiolactone.

The author confines the discussion to the fibers in Section II, and selects nylon, Dacron, and Orlon as representatives of the three subclasses. He lists the physical properties of these three fibers as follows:

Nylon: high strength, high elasticity, low modulus, high abrasion resistance, delayed stretch recovery, good alkali resistance, moderate acid resistance, good flame resistance.

Dacron: high strength, moderate elasticity, high modulus, high resilience, more rapid stretch recovery, moderate abrasion resistance, moderate alkali and acid resistance, moderate flame resistance.

Orlon: moderate strength, good elasticity, high flex resistance, high bulk density, excellent acid resistance, moderate alkali resistance, excellent sunlight resistance, excellent hand.

Nylon appears to be well established as a knitting fiber where its strength, elasticity, and low modulus combine to give it advantages over any other fiber presently known. In certain industrial applications, such as in ropes and fish nets, the only factor which appears to hinder its almost universal adoption would appear to be in the cost field. The use of nylon where abrasive conditions are se-

vere is obviously on the increase and will expand.

Dacron is a much more recent arrival. It seems likely that in certain applications, such as worsted-type suitings, particularly in the relative light weights, this new fiber will find a very high place due to its resilience and its properties of shape retention. In continuous filament form, time may prove that this material possesses advantages in broad-woven fabrics similar to those which have been proved for nylon as a knitting fiber. In some twine and cordage applications as well as in sewing thread it may succeed where the excessive stretch involved in nylon's low modulus has been a disadvantage to date.

Orlon too is relatively new and the field of its application is yet to be fully explored. Outstanding factors appear to be its high bulk density and its warm soft hand. Combined, these properties would appear to give potential advantages to the fiber in winter clothing, including sweaters and lining fabrics. Orlon is claimed to be the most wool-like of the synthetics when in the crimped staple form, and the most silk-like when used as a continuous filament. We may expect, therefore, that in blankets, overcoats, and dress goods the various forms of Orlon will find their uses. Its great sunlight durability indicates ultimate acceptance in tents, awnings, and other outdoor uses where cotton ducks and canvases now hold sway.

One type of application which we are just beginning to observe is the blending of synthetic fibers with the already well-established rayons, acetate, and natural fibers. To these blends each of the fibers listed above will bring its own properties—nylon for strength, Dacron for resilience, Orlon for warmth, etc.

The author discusses the efforts being made by chemists throughout the world to develop still other new fibers. While not attempting to predict the properties of these future products, he states that there is a definite need of new fibers possessing the following properties:

(1) A truly synthetic fiber retaining the advantages of wear and freedom from biological attack but which will also felt or mill.

(2) A synthetic fiber that will give substantial lateral swelling when wetted. True waterproofness is very difficult to obtain with a nonswelling fiber, and this development would be of great assistance in these fields.

(3) A fireproof fiber with a full range of colors. Nylon and Dacron are flame-resistant but can be destroyed by fire. A modified form of Orlon exists that is essentially fireproof, but due to molecular rearrangement of the material, which allows free carbon to form in the fibers, it is only available in black.

(4) A truly elastic fiber combining the resilient properties of rubber with the durability of the synthetic textiles would be a great boon to the makers of all elastic fabrics.

The textile industry got along with four fibers for more than 5000 years, yet within a single generation we have multiplied this number by three or four. The next generation may multiply it by a hundred.—WHC

Dyes and Dyeing; New Man-made Fibers—New Problems

Anon, *Textile Mercury & Argus* **126**, 202, 205, February 1, 1952.

Experiments now being made by dyestuffs manufacturers in the dyeing of the newer man-made fibers are opening up exciting possibilities of new techniques which may mean, in a few years' time, radical changes in textile dyeing methods and equipment.

The main problem facing the dyestuffs industry is that the latest man-made fibers, such as Terylene (Dacron) and Orlon, which have high strength and wearing qualities, require dyeing to a fastness standard that will outlast or equal the life of the fabric. To achieve this, dyestuff chemists are beginning to favor high temperature pressure dyeing. To do this commercially, new dyeing equipment will be needed in the dyeing industry. The advantage of high temperature dyeing is its speed and saving of processing time.

As Terylene and Orlon are both highly orientated fibers, it is difficult for large dyestuff molecules, which are essential for highest fastness properties, to penetrate into these fibers under normal dyeing conditions. Dyeing assistants or "carriers" can be used to increase the uptake of dye and to swell the fibers to permit diffusion to take place more rapidly, but only by very high pressure dyeing is it possible to make the large dye molecules penetrate these highly orientated fibers.

Experiments made at the ICI laboratories in dyeing Terylene with dispersed acetate dyes show that, at a temperature of 125-130° C, the dyeing becomes extremely rapid and there is no difficulty in getting high exhaustion of the dye into the fiber. It has also been found that once acetate dyes are dispersed into Terylene they are much faster to wet treatment than they are on nylon or acetate rayon.

It is to avoid this very high temperature dyeing (125-130° C) that fiber swelling agents are being used, as there is no pressure equipment in the dyeing trade capable of dyeing fibers at these high temperatures.

In addition to pressure dyeing, two other methods that lend themselves to the dyeing of the newer synthetic fibers in a much shorter time than the normal methods are being investigated by ICI. They are the pad-steam process and the pad-dry heat process.

The application of the pad-steam technique to Terylene has shown promise, provided that steaming is carried out under pressure; for example, in a cottage steamer.

The pad-dry heat process consists of padding the fabric through a concentrated aqueous dispersion of dyestuff, drying it, and then subjecting the material to dry heat at a high temperature for a short time. The dispersed acetate dyes are applied successfully by this process, with a heat treatment of 230° C for 10 seconds. The writer comments, however, that Terylene tends to shrink at these high temperatures if not held in tension.—WHC

Carpet Mills, the Fibers and the Future

J Campbell, *Rayon & Synthetic Textiles* **33**, 31-2, 97, April, 1952.

During the past two years, the impact of chemical fibers has revitalized an industry previously confined chiefly to wool. From now on, the soft floor covering manufacturers will use rayon, acetate, wool, cotton, nylon, Saran, dynel, and perhaps others, wherever these fibers will serve to make more attractive and durable floor coverings.

Two years ago the carpet mills realized they would have to break their dependence upon wool or face a disastrous decline in sales and profits. One by one, the major producers brought out fabrics of wool blended wtih rayon and acetate, as well as all-synthetic floor coverings. Since then, virtually every carpet manufacturer has increased his use of man-made fibers. At present, rayon and acetate are as important in the carpet industry's scheme of things as fibers clipped from sheep. The use of cellulosic fibers during 1951 was approximately 24 per cent of the total fibers consumed by the industry for surface yarns.

The manufacturers now realize that they can make better carpets with man-made fibers. It is this growing conviction that is providing the energy for the current revitalization of the carpet industry.

Even in the field of carpeting for theaters, hotels, restaurants and other public places, known in the industry as "contract business", the new carpets are making progress. The manufacturers have been able to convince hard-boiled purchasing agents that the man-made fibers can stand up under the strenuous wear that contract carpets must take.

The writer cites several examples of the new floor coverings. One mill is making a carpet with an all-rayon surface; another produces an all-acetate face. Blends of wool with rayon, Celcos, nylon and vinyl yarns are described. One of the New York hotels now has a large carpet made with a blend of wool and Avisco 15 carpet rayon.

The writer asks, "Will the carpet industry progress to a 100 per cent chemical fiber basis?" He believes that such a development is unlikely.—WHC

The Acid Exhaustion-Thermosol Process for the Dyeing of Fabrics Made from Dacron Polyester Fiber

Anon, *Technical Bull* (*Du Pont*) 7, 198-202, December, 1951.

The acid exhaustion-Thermosol process represents a suitable method for dyeing filament and staple fabrics made of Dacron in light to medium shades with selected dispersed colors by a simple two-step procedure. The first step involves the use of a small amount of an inorganic or aliphatic organic acid to exhaust the color from an aqueous bath onto the fiber; the second step entails heating the fabric for a short time to fix the color within the fiber.

The new process is particularly well adapted to the coloration of small yardages of cloth. It avoids the disadvantages of carriers and does not require the use of pressure to achieve color fixation.

Fabric made of Dacron in filament or staple form is dyed in an aqueous bath at temperatures of 205-212° F, using 1.5 to 5 grams per liter of sulfuric, formic, acetic, or chloroacetic acid to exhaust the color onto the fabric. Sulfuric acid is most commonly used, and the addition of a salt is sometimes helpful.

Filament fabric is best dyed in a covered stainless steel jig containing sufficient steam lines above and below the surface of the bath to maintain a temperature of 211-212° F. Staple fiber is dyed in a closed stainless steel beck having sufficient heating capacity to maintain a temperature of at least 205° F, preferably 211-212° F.

Color development is attained by subjecting the dyed fabric to a short heat treatment at temperatures of 360-440° F. The Thermosol process brings about complete penetration of the colors into the fiber, and produces dyeings that possess the maximum fastness properties of the dyes used.

The choice of dyes for use in this process should be based on their affinity for Dacron in the presence of dilute acid, on their leveling properties, and on their ability to withstand the Thermosol fixation conditions. A selected list of dispersed acetate dyes is recommended. —WHC

Optical Bleaching Agents

C R Trotman, *Textile J Australia* 26, 1224-7, February, 1952. (from *Hosiery Trade J*)

Fluorescence is a color phenomenon differing fundamentally from that shown by dyestuffs. A fluorescent substance is one which absorbs rays of light of one color and re-emits them again in a different color, that is, of a different wave-length. Thus a fluorescent surface, unlike one which is dyed, does not subtract from the light which falls on it, but only alters its nature. This property of fluorescence is being used to an increasing extent in the production of whites, since certain fluorescent substances will absorb invisible ultraviolet light, a little of which is present in ordinary daylight. This ultraviolet light is re-emitted as visible light, usually at the blue or violet end of the spectrum. When applied to bleached textiles, such substances neutralize the slight residual yellow tint, not by subtracting from the reflectance as dyes and pigments do, but by adding to the total light reflected, since the absorbed light is in the invisible region. In this way whites of an intensity hitherto unknown are created, and by comparison the whites to which we are accustomed appear lifeless.

There seems to have been more readiness to use these new compounds with cellulosic fibers than with others. This is partly because the earliest products marketed were specifically for cellulose, and partly because a warmer or creamier tint is associated with wool.

The fluorescent compounds recommended for cotton and other cellulosic fibers resemble the direct dyes in composition, with the exception that the conventional chromophore or color-producing constituent is absent. They are therefore, in effect, colorless direct dyes and behave as such. They are applied from a dyebath with the usual liquor ratio of 20:1, and better exhaustion is obtained if salt is added.

It must not be assumed that optical bleaching agents are a substitute for ordinary bleaching, for they have no such properties of their own in the normal sense. They can be used with success only to create an entirely new standard of whiteness on goods which have already been bleached, or to make the color of a mild bleach comparable with that of a full one. There is an important point to be borne in mind, for the white produced by the optical bleaching agent is not permanently fast to light, and after prolonged exposure the ultimate shade will be that of the base to which it was applied.

The use of these fluorescent substances is no less important in dyeing than in bleaching, and if they are used judiciously they can brighten and enhance the appeal of many shades. As would be expected, this applies mainly to pas els and some of the brighter colors for knitted outerwear. There are many of these substances available, and the best results can only be obtained if the right one is selected.—WHC

NEWS

• V-C Chemical Opens New York Office

Several hundred representatives from the textile industry visited the new facilities of the Virginia-Carolina Chemical Corporation at 500 Fifth Avenue, New York during the official opening on April 17th.

The New York office, of which Lee B Whitcomb will be in charge, represents a further step in the expansion of V-C's new Vicara zein fiber.

On hand during the opening ceremonies were members of the Corporation's board of directors (including Joseph A Howell, president, Irving D Dawes, vice-president and treasurer, and Charles E Heinricks, vice-president) as well as other representatives from V-C's general offices in Richmond, Va (including John H Karrh, manager of the firm's Fiber Division) and Jesse F Yeates, Jr, sales manager).

Among the more than 125 representatives of the woolen textile industry who attended the Wool Research Project Seminar, held at Lowell Textile Institute, Lowell, Mass, April 7-9, were several prominent international scientists. Disclosures of the work done in the International Wool Research Project were made at the seminar, which was held under the auspices of The Wool Bureau, Inc. Pictured left to right, standing, are: Dr E G Carter, Director of Science and Technology, International Wool Secretariat, London; Dr Gösta Silen, Research Director of The Finnish Wool Industries Research Laboratory; Birger Eriksson, Director Swedish Institute for Applied Textile Research, Sweden and Giles E Hopkins, Technical Director, The Wool Bureau, Inc. Seated, from left to right are Dr John H Dillon, Director, Textile Research Institute, Princeton, N J, and Werner von Bergen, Director of Research, Forstmann Woolen Company.

• Gordon Research Conferences

Facilities at Colby Junior College, New London, N H, have been made available for the annual Gordon Research Conferences from June 16 to August 29. The Conferences, sponsored by the American Association for the Advancement of Science, were established to stimulate research in universities, research foundations and industrial laboratories.

The meetings are of the informal type, consisting of scheduled lectures and free informal discussion groups. Meetings are held in the morning and evening, Monday through Friday, with the exception of Friday evening. Afternoons are available for reading, recreation, participating in discussion groups, etc.

The purpose of the program is not to review the known fields of chemistry, but primarily to bring experts up to date as to the latest developments, analyze the significance of these developments, and to provoke suggestions as to underlying theories and profitable methods of approach for making new progress. In order to protect individual rights and to promote discussion, it is an established rule of each Conference that all information presented is not to be used without specific authorization of the individual making the contribution, whether in formal presentation or in discussion. No publications are prepared as emanating from the Conference.

Individuals interested in attending the Conferences are requested to send their applications to the Director on or before May 15th. Each applicant must state the institution or company with which he is connected and the type of work in which he is most interested. Attendance at each Conference is limited to 100. Accommodations are available for a limited number of women to attend each Conference, and also for wives who wish to accompany their husbands.

Requests for attendance or additional information should be addressed to W George Parks, Director, Dept of Chemistry, Univ of Rhode Island, Kingston, R I. After June 15 mail should be addressed to Colby Junior College.

Following is the program for the 1952 Conference on Textiles, of which W J Hamburger is Chairman and J Edward Lynn, Vice-Chairman:

July 14—

Herman F Mark. "New Polymers for the Formation and Modification of Textile Fibers".

Bruno R Roberts. "Influence of Some Colloid Chemical and Physical Factors on Textile Performance of Synthetic Fibers, Especially of the Acrylonitrile Type".

July 15—

Kenneth L Hertel. "Response of Fiber Assemblages to Compression as Influenced by Certain Fiber Properties".

A G H Dietz. "Transmission and Attenuation of Energy in High Polymers".

July 16—

Richard O Steele. "The Relationship Between the Chemical Structure and the Mechanical Behavior of Wool".

Turner Alfrey, Jr. "The Relationship Between the Chemical Structure and the Mechanical Behavior of Synthetic Fibers".

July 17—

Harold P Lundgren. "Action on Wool of Certain Low Molecular Weight Reagents".

Henry M Morgan. "Effects of Chemical Treatments on the Mechanical Behavior of Human Hair".

July 18—

Orville C Wetmore. "The Effects of Various Fabric Types on Body Comfort as Measured by Physiological Responses of Human Subjects".

• Monsanto Establishes Fellowship at N C State

The establishment of a Monsanto Chemical Company fellowship for research in textile chemistry and physics at the North Carolina State College, Raleigh, N C, was recently announced by E G Somogyi, Merrimac Division research director.

The fellowship will reportedly run for one year supported by a $1200 contribution. Under the terms of the agreement with North Carolina State, the fellow will be chosen by the Textiles and Graduate Schools of the College. While the fellowship will be for graduate work in the School of Textiles, it will be open to qualified students with degrees in chemistry, chemical engineering, mechanical engineering, physics or other scientific and engineering courses and will be awarded on the basis of outstanding achievement and promise, it is learned.

• New Grade Cuts Cost of Dimethylethanolamine

A new low-priced, industrial grade of Dimethylethanolamine, recently announced by Carbide and Carbon Chemicals Company, is being offered at 52.25 cents per lb (of concentrated amine) in tank car quantities. The new material, "Dimethylethanolamine, 70%," is a 70 per cent solution of the amine in water, designed for use in the manufacture of ion exchange resins, wetting agents, textile assistants, etc.

• Symposium on Newer Synthetics Well Attended

A symposium on "The Status of Newer Synthetic Fibers", sponsored by the Chemical Marketing Subdivision of the American Chemical Society during that organization's recent national meeting, attracted a large crowd to hear seventeen papers by outstanding textile authorities.

Carl A Setterstrom, Carbide and Carbon Chemicals Company, acted as Chairman of the two-day symposium. F A Soderberg, General Dyestuff Corporation, heads the Chemical Marketing Subdivision.

The selection of the fibers from which new textiles are made, the effect of their chemical structure on fabric properties, and the comparison of the strength of new textiles were the subjects of the initial morning session of the symposium. Dr Winfield W Heckert of the textile fibers department, E I du Pont de Nemours & Company, Inc, spoke on the problem of choosing from many chemical sources a fiber whose advantages will be most useful in a diversity of textile applications. Professor Herman F Mark, director of the Polymer Research Institute, Polytechnic Institute of Brooklyn, described the important chemical criteria of fibers, and Dr J H Dillon, director of the Textile Foundation and Textile Research Institute, Princeton, N J, compared the mechanical properties of textiles from various starting materials.

The performance characteristics of new fabrics, the cost and availability of raw materials, and the factors which influence choice of location for a textile plant were outlined by Rene Bouvet of the textile research department, American Viscose Corporation, Marcus Hook, Pa, Howard Bunn, vice president of the Carbide and Carbon Chemicals Company, and C O Hoyer, director of engineering of the Chemstrand Corporation, Decatur, Ala, in the Wednesday afternoon session, which was devoted to these and other production problems.

The impact of the new fibers on the markets for natural fibers and the older synthetics constituted the somewhat controversial subject-matter of the Thursday afternoon session. Dr Werner von Bergen, director of research of the Forstmann Woolen Company, Passaic, N J, spoke on the markets for wool and for wool-synthetic blends, and L K Fitzgerald, director of research of Dan River Mills, Inc, Danville, Va, described the effect of the new synthetics on markets for cotton, the "work-horse" fiber of the textile industry.

The present position of the silk market and the future possibilities of silk were covered by W S Kilborne, vice president of the William Skinner & Sons Company, New York.

Julius B Goldberg, J P Stevens & Company, Inc, New York, discussed rayon; Ashton M Tenney, A M Tenney Associates, Inc, New York, covered the field of acetate fiber.

F A Soderberg

A paper on the "Functionally Significant Properties of the New Synthetics" by Joseph B Quig, E I du Pont de Nemours & Company concluded the program.

• Avisco Simplifies Process for Dyeing Acetate Tricot

Slides and large-scale drawings at last month's Knitting and Allied Crafts Exposition displayed a continuous process for dyeing acetate tricot as developed by American Viscose Corporation. While cutting costs, the process is said to improve quality and improve shrinkage.

The system reportedly presets and dyes the fabric at the same time in open-width form, eliminating the necessity for tacking and untacking and reducing the dyeing method to two steps.

Tests made by Avisco's Textile Research Department at Marcus Hook, Pa, are said to indicate an increase of fabric strength of at least 10% when compared to dyeing in the conventional rope form.

• New Cyanamid Subsidiary Formed

K C Towe, President of American Cyanamid Company, has announced the recent formation of a new corporation, Chemical Construction (Inter-American) Ltd. A Cyanamid subsidiary, with main offices in Toronto, Canada, Chemical Construction (Inter-American) Ltd will reportedly design and build chemical plants in all nations of the Western Hemisphere except the United States. Its operations will parallel those of another Cyanamid unit, Chemical Construction Corporation.

President of the new company is W R Geddes, Assistant to the President of North American Cyanamid Limited, which is wholly-owned by American Cyanamid Company.

• Foxboro Expands Southern Service

With the addition of new branch offices in Decatur, Ala, and Greenville, S C, as well as the appointment of additional personnel, The Foxboro Company, Foxboro, Mass, has further extended its sales and service facilities to the South.

W W Barron is in charge of the Greenville office while C A Mohler heads the Decatur branch. The new branches bring the Foxboro total in the region to six, the others being at Atlanta, Birmingham, Jacksonville and Charlotte.

E W Prendergast has moved up to branch manager at Atlanta, succeeding Mr Barron. Added to the Atlanta staff is F H Feathers.

Birmingham has added a new engineer, L B Perry, working with the Branch Manager, J M Waters. The Jacksonville Office is now under the direction of R M Schneble, assisted by J L Pierce. The Charlotte office, under the direction of S A Alexander, has announced the appointment of H A Ruddock as sales engineer.

All of the Southeastern area comes under the supervision of A H Shafer, Assistant Division Manager, Eastern Sales Division, whose headquarters are at Pittsburgh, where The Foxboro Company has a branch factory and warehouse.

• Electron Microscope Course

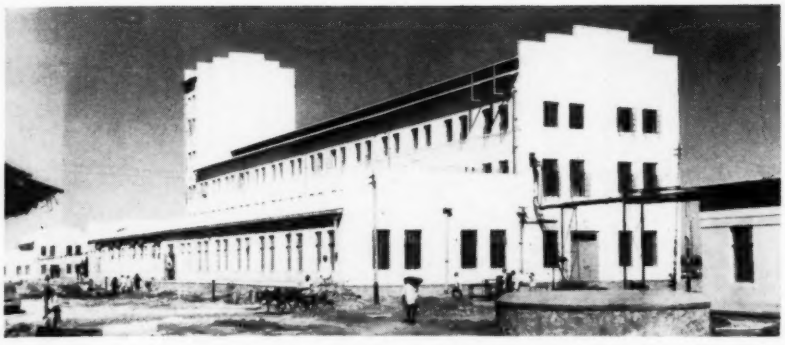
Cornell University will again sponsor a summer laboratory course in Techniques and Applications of the Electron Microscope from June 16-28. The course will be given by the University's Laboratory of Electron Microscopy in the Department of Engineering Physics. Dr James Hillier of the RCA Laboratories, Princeton, N J, and Dr C E Hall of MIT will be guest lecturers.

The course is designed for those research workers, institutional and industrial, who have recently entered the field of electron microscopy or who are now planning to undertake research problems involving applications of this instrument.

Further inquiries should be addressed to Dr Benjamin M Siegel, Department of Engineering Physics, Rockefeller Hall, Cornell University, Ithaca, New York.

• Hooker-Sponsored Fellowship

Hooker Electrochemical Company is now sponsoring a research fellowship in chemical engineering at the University of Washington. While no specific assignment has been made, the research reportedly will be concerned with uses of caustic soda and chlorine as related to the use of waste products of the pulp industry such as lignin.



Exterior view of the azo and sulfur dyes plant of the newly-formed Atul Products Ltd, at Bulsar, India.

• India Looks to New Bulsar Plant for Dyestuffs

With the opening of a $3,500,000 dyestuff and pharmaceutical plant at Bulsar (150 miles north of Bombay) last March, the Indian textile industry—long dependent on imported dyestuffs—now looks to its own source of supplies.

The new company, known as Atul Products Ltd, is a result of a merger of Indian capital and technical "know-how" of the American Cyanamid Company. Under the arrangement, 90% of Atul's capital is subscribed by India's bankers and businessmen. American Cyanamid has reportedly put up 10% at the request of the Indians as a token of its interest in Atul's 20-year program of development. Initially, Cyanamid has offered technical assistance, personnel and raw materials.

First product to leave the blenders in the new plant is sulfur black, forerunner of an initial range of 25 of the dyes most widely used in India. Azo dyes, however, will constitute most of the output, it is learned. The main part of the dyes section of the factory is a three-story structure, surmounted at one end by a 90-foot tower that houses the ice plant. Production capacity of this building alone is reportedly rated at 4,000,000 pounds per year—one million of sulfur dyes and three million of azo.

The method of manufacture will be in general the same as that employed at the Bound Brook plant of the Calco Chemical Division of Cyanamid. The Atul project will also have the continuing benefit of laboratory research conducted at Bound Brook. At the same time, Atul already has its own laboratory, which will handle specific dye problems posed by Indian customers.

The man who is pioneering the evolution of the new Indian dyes industry, Kasturbhai Lalbhai, reportedly owns the biggest group of cotton mills in Western India. He also sits on many company boards in other industries, regularly leads Government Trade Delegations and heads national committees.

The laboratory and the plant are manned by Indian chemists (some with American college degrees) and trained workers from nearby villages. The man in charge of azo and sulfur dye production is S S Desai, an Indian chemist who worked for 15 years in the Kasturbhai group of mills as consultant on bleaching. Last year Mr Kasturbhai sent him to Bound Brook for five months, and now the man who trained him there, Cyanamid's George French, is at Atul on a six-month assignment "to see the new plant off to a good start." Since August of 1951 Mr French has personally supervised the installation of equipment and the training of local technicians.

The lay-out of the plant, is said to allow maximum efficiency in handling of materials. The 40 tons of ice and ten tons of refrigerated water required daily by the azo section are supplied by an automatic ice-making machine (Henry Voght of Philadelphia), assisted by a barometric leg vacuum cooler. The ice is reportedly fed by gravity to the solution vats on the third floor in which the initial solutions are made up. These are again fed by gravity to the huge 10,000 gallon coupling vats which are set just above floor level on the second story.

All the vats in the plant have been made on the spot by Indian craftsman shipbuilders with Indian teakwood cut from the local jungles. The chemical reaction of teakwood to the various acids and alkalis used in dye manufacture is as yet unknown, and Atul chemists will be watching to test the life of the teakwood as against the cypress usually employed in the United States. Teakwood manufacture has cut costs by about half, it is stated.

After leaving the coupling vats, the precipitated dye flows by gravity to centrifugal pumps which convey the dye slurries to the large-size (56-inch) plate-and-grame filter presses. The isolated press cake is then loaded into trays and dried in recirculating air truck driers. Finally the dye is dumped into containers and carried back to the third floor where it feeds down through the grinders to the blenders and to the finished packages ready for shipment.

The sulfur group of dyes, which are made in the same building at the end remote from the ice-tower, use similar grinding and blending equipment, but are dried on drum driers.

Cyanamid experts who have helped to build Atul emphasize that production possibilities extend far beyond present capacity. A natural development will be production in India of some of the crudes. Benzol, for example, is already made by an Indian firm.

As to size—there is plenty of room for expansion in Atul's 850 acres.

• ASQC to Meet in Syracuse

Better than 2000 persons are expected to attend the Sixth Annual Convention of the American Society for Quality Control to be held May 22-24 in Syracuse, N Y. Convention headquarters will be at the new Onondaga County War Memorial Auditorium. A young and vigorous group, the ASQC consists of inspection, production, management and educational leaders.

Under the general chairmanship of Leon Bass, General Electric Co, the technical program has been planned to provide papers and discussions in 18 fields, including textiles and textile machinery, chemical, management, pulp and paper, electronics, etc.

Numerous exhibits will supplement the Convention.

• New Milliken Film

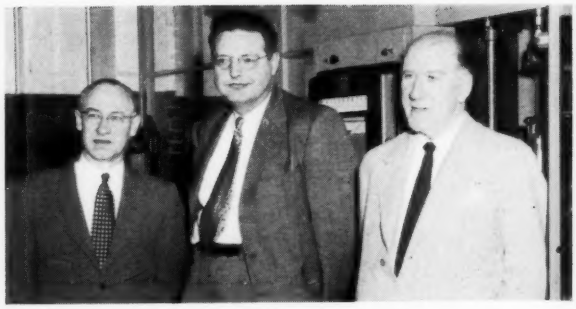
"Fabric Magic" is the title of a 20-minute color picture recently completed at the studios of The Princeton Film Center, Inc for Deering, Milliken & Company. The picture shows the results with which Milliken is now blending the new synthetic fibers, Orlon, Dacron and nylon, into wool to obtain a wrinkle proof fabric of great utility.

"Fabric Magic" was produced primarily for showings to retail sales personnel, but prints are reportedly available to schools, clubs and other interested organizations.

• USICC Relocates

U S Industrial Chemicals Co, Division of National Distillers Products Corporation, has announced the relocation of its head office, formerly at 60 East 42nd Street, New York 17, N Y. The new address is 120 Broadway, New York 5, N Y. The move does not affect the address of USI's New York Sales Division offices, which remain at 33 West 42nd Street, New York 18, N Y.

Shown above are garments and materials that have been "Nylonized" by a new and exclusive finishing process developed by Hans C Bick, Inc, Reading, Pa. The process, in which Type 8 nylon is bonded to the yarn comprising the fabric, is said to make nylon, acetate, or rayon softer, longer wearing and more absorbent. Other features include its relative ease of application, its economy, its comfort and its promotional advantages. These and other nylonized garments were exhibited by Bick at the recent Knitting and Allied Crafts Exposition in New York.



Members of the textile industry serving as advisers to the Cotton Fiber Division of the Southern Regional Research Laboratory held their annual conference with the scientists in New Orleans recently. Left to right: Wayne A Sisson, American Viscose Corp; Harold M Spurlin, Hercules Experiment Station, Wilmington, Del; Emil Heuser of La Jolla, Calif (formerly with the Institute of Paper Chemistry, Appleton, Wis). Reports were heard on the Laboratory's projects on "decrystallization," flameproofing, chemical modification, and microscopical properties of cotton fiber.

• First Licenses to Use Avcoset Process Signed

First licenses for the use of Avcoset, American Viscose Corporation's process for shrinkage and wear control, were signed on April 16 with Julius Hermes, manager of the Fontaine Converting Works, Martinsville, Va, signing as the first finisher and Harold Freedman, president, and Edward J Bartmann, treasurer, of Lesem Bach & Co, New York, as the first converter.

Arthur R Wachter, manager of Avisco's converting relations department, signed for the corporation. Also present at the ceremony in the Avisco board room were Harry L Dalton, vice-president in charge of sales and public relations; George L Storm, manager of rayon staple sales, and the following representatives of the corporation's Textile Research Department, Marcus Hook, Pa, developers of the process: Edgar S Kennedy, director; Arthur N Tingley, head of the textile chemicals division; Jackson A Woodruff, head, and Irvin H Welch, Jr, of the dyeing and finishing division.

• Speakers at TI Conference

Among those scheduled to deliver papers at the Textile Institute's 37th Annual Conference (Edinburgh, Scotland, June 2-6) are E S Kennedy, American Viscose Corp; Lloyd L Leach, E I du Pont de Nemours & Co, Inc; Professor F Maillard, Institut Textile de France; H Nuding, Phrix, Germany; and representatives of Vezelinstituut TNO (Holland), the Societe Rhovyl, and the U S Navy, as well as British research associations, Leeds University and leading concerns such as Imperial Chemical Industries, L d, TMM (Research) Ltd, British Celanese Ltd and British Nylon Spinners Ltd.

The subject will be "Fiber Blends."

• Barrington Engineering Moves Executive Offices

The Barrington Engineering Corporation, manufacturer of high-speed wet mixing equipment of the turbine type, has moved its executive and sales offices to 110 W 40th St, New York 18, N Y.

• C & A to Award 4-Year Scholarships

Four-year scholarships, three to be awarded each year, have been granted to textile schools in plant areas by Collins & Aikman Corporation, it is learned.

Albert R Jube, president, has stated that the scholarships are to be used primarily for employees' children. However, he added, the scholarships would be awarded to the community if, in any year, there are no eligible applicants from the families of employees. He pointed out that within four years, twelve students would be matriculating under the company's program.

The purposes for granting the scholarships were reportedly to provide additional education to children of employees, to support local textile schools, and to serve the textile industry by encouraging promising young men and women to enter the field.

The schools receiving the grants are the North Carolina State College, School of Textiles; the Philadelphia Textile Institute, and the Rhode Island School of Design.

NAMES IN THE NEWS

CHARLES F McKENNA, JR, a veteran of 30 years in the organic chemical and process industries, has established an office at 2300 Connecticut Avenue, Washington, D C, where he will represent industrial groups operating in the chemical and chemical process industries.

Mr McKenna served as Washington representative for the past year for all divisions of General Aniline & Film Corporation.

Prior to joining General Aniline in 1945, Mr McKenna was with Commercial Solvents Corp for 17 years in the market development and sales departments.

HERBERT J HEESCH has been made field sales supervisor of Hooker Electrochemical Company. In his new position, Mr Heesch will be responsible for coordinating the activities of field salesmen and district sales managers. He will make his headquarters at Niagara Falls.

LESTER O PFAHL has joined Hooker as a mechanical engineer in the operations department. BRANTFORD H DIETRICH has been employed as a plant engineer and ARTHUR S COOKFAIR has become a laboratory assistant in the research department.

R P Parker

DR ROBERT P PARKER was named director of chemical research of the American Cyanamid Company, Calco Chemical Division to succeed DR H Z LECHER following his retirement on April 1.

Dr Parker came to Calco as a student trainee and joined the Research Department in 1934. He became a group leader in 1938 and sectional director in the Organic Research Section in 1944. During this period, his work was mainly in the field of dyes and pigments.

Dr Parker was appointed assistant to the director of research in 1946 and became assistant director of research of the Pharmaceutical Section in 1947.

Henry M Kamins

HENRY M KAMINS will represent Paco Textiles (New York) throughout New York and New England in his new capacity as assistant sales manager.

Paco Textiles is the national sales representative for four affiliated North Carolina textile firms, including Central Yarn and Dyeing Company, of Gastonia, N C, dyers of cotton and synthetic yarns.

This new post represents a return to this organization for Mr Kamins, because he served as a production and cost analyst for the Central Yarn and Dyeing Company before World War II.

HUBERT A DesMARAIS has been appointed general sales manager of the Pennsylvania Salt Manufacturing Company of Washington.

Mr DesMarais, formerly Pacific Coast sales manager for the General Dyestuff Corporation with headquarters in San Francisco, will locate at Pennsalt of Washington's central office in Tacoma.

Mr DesMarais came to Portland, Oregon, in 1932 to open and manage the first General Dyestuff Corp office there. In 1937 he was appointed Pacific Coast manager. Previously he had been technical and sales representative on dyestuffs and chemicals for the paper board and pulp mills in New England. Before joining General Dyestuff he was in the production department of the Chemical Paper Manufacturing Co, Holyoke, Mass.

Mr DesMarais served as the first secretary of the Pacific Coast Section in 1949 and chairman of the Pacific Southwest Section in 1950.

A N De Vito

ANTHONY N DE VITO completed his first year as superintendent of dyeing and finishing at Tex-Lane Dyeing Co, Inc of Brooklyn, N Y on April 23. Tex-Lane, a relatively new firm, does piece goods dyeing and finishing in the outerwear trade.

Mr De Vito, a member of AATCC since 1932, has had extensive experience in dyeing, flameproofing, water repellency and residual finishes. Prior to joining Tex-Lane, he was superintendent of finishing at Antipyros Co, Inc.

COLONEL CHARLES F H JOHNSON, president of Botany Worsted Mills, will be the principal speaker at Commencement Exercises of Philadelphia Textile Institute, Philadelphia, Pa, on June 7th.

Colonel Johnson has been associated with Botany Worsted Mills since 1920 and president of the company since 1929. He served as a Major in the Air Force during World War I, and as a Colonel in the Air Force as advisor to Director of Army Emergency Relief in 1942.

MORSE G DIAL has been elected president of Union Carbide and Carbon Corporation, suceeding FRED H HAGGERSON, who continues as chairman of the board for UC&CC. KENNETH H HANNAN has been elected treasurer, and WALTER E REMMERS, vice president—Alloys Division.

Mr Dial has served as executive vice president of Union Carbide since 1951. He joined the Corporation in 1929.

C Kerns Batson

C KERNS BATSON has been appointed sales representative for Virginia, North Carolina and part of South Carolina for the Augusta Chemical Co, Augusta, Ga.

Mr Batson has been connected with the dyeing and finishing industry for the past 18 years and has served as engineer in the construction of plants and the installation of equipment for such companies as United Merchants and Manufactures, Inc, Dan River Mills, and North Carolina Finishing Co.

J C WHITT has assumed the newly-created position of assistant superintendent in charge of dyeing at the National Plant of Standard-Coosa-Thatcher Company, Chattanooga.

Mr Whitt's varied experience in dyeing and processing the newer synthetics stems from sixteen years in the field. He was first employed by Cloverdale Dye Works, Inc in his native High Point, N C, in 1936. He later became superintendent of dyeing at Morgan Cotton Mills, Inc, Laurinburg, N C prior to service with the Chemical Warfare Division, AAF, during the war. In 1946 Mr Whitt was employed to supervise the building of a yarn dyeing plant for Standard Hosiery Mills, Inc, Burlington, N C, and later became superintendent of dyeing and winding upon its completion, a position he held until joining Standard-Coosa-Thatcher.

Mr Whitt has held the offices of sectional committeeman, treasurer and vice-chairman of the Piedmont Section, AATCC.

C W Gayler

L E Dequine, Jr

Louis F Ray

Paul W Runge

ROY G HEMMINGHAUS, manager of The Chemstrand Corporation's nylon plant now under construction at Pensacola, Florida, last month announced four appointments to key positions in nylon manufacturing and process operations: DR C W GAYLER, technical superintendent, L E DEQUINE, JR, plant engineer, LOUIS F RAY, superintendent of intermediates, and PAUL W RUNGE, chief chemist.

All have been engaged in early phases of the nylon research and organization activities since the company was licensed by E I du Pont de Nemours & Company, Inc to manufacture and market nylon filament yarn. The nylon plant, which is being designed and built by Du Pont on a 2,000 acre site north of Pensacola, will reportedly have a 50,000,000 pound per year capacity. Production is expected to begin in late 1953.

WALTER R SHEPPERD, who is retiring after forty years of service with Stein, Hall & Company, Inc, New York, was tendered a farewell luncheon on April 23 by many of his fellow workers. Morris S Rosenthal, president of the company, thanked Mr Shepperd for the loyal and long service he had given to Stein-Hall and presented him with a sterling silver cigarette box, commenting that not only Mr Shepperd's friends at Stein-Hall but also the many business associates with whom he had dealt for so many years wish him well in his retirement.

Mr Shepperd started with Stein-Hall in 1912 and was a salesman in the Company's Eastern Territory. Mr and Mrs Shepperd plan to move from New York City to a new home somewhere in the South, it is learned.

ELMER M MATTER, former district manager at Grand Rapids, Mich, for the Procter & Gamble Company, has been named chief of the glycerine, soaps and detergents section of the National Production Authority's Chemical Division.

Mr Matter succeeds GEORGE A WRISLEY, SR, who has resigned to return to his position as vice-president of the Allen B Wrisley Co, Chicago, Ill.

NEW members of American Cyanamid Company's New Product Department include DR J G AFFLECK and DR J C PULLMAN.

Dr Affleck, with the Cyanamid organization since 1949, was formerly employed in the Stamford Research Laboratories in process development work.

Dr Pullman, who was also at the Stamford Laboratories formerly, joined Cyanamid in 1943.

HERBERT R SCHWARZ now heads textile research activities at Wellington Sears Company in the newly-created position of director of research and fabric development. He has been active in the development of both cotton and synthetic industrial fabrics since joining Wellington Sears in 1935.

SIDNEY FISHMAN, RICHARD STEELE, and AARON WEINSTEIN, formerly of Heineman & Seidman, have entered their own textile brokerage business at 141 West 40th St, New York 18, N Y. The new firm, Fishman, Steele & Weinstein, is dealing in rayon and synthetic fabrics and yarns.

OBITUARY

C IRVING LAKEY

C IRVING LAKEY, a representative of Jacques Wolf & Company for the last 14 years, died on April 15 after a short illness.

Mr Lakey, a resident of Smithfield, R I, was formerly boss dyer for the Cooper-Kenworthy Co of Warren and the Eagle Dye Works and the Eureka Dye Works of Pawtucket.

He was a member of the Rhode Island Section, AATCC.

C LAURENCE WARWICK

C LAURENCE WARWICK, executive secretary, American Society for Testing Materials, and its administrative head since 1919, died suddenly Wednesday evening, April 23, shortly after presiding at a dinner honoring the retiring treasurer of the Society. During the meal he complained of chest pains and after taking a sedative, continued, but died about an hour after the close of the affair.

Mr Warwick, who was born July 29, 1889, had been active in ASTM since 1909. He had made many notable contributions to the field of standardization and research in materials and was recognized as an outstanding authority on materials. One of his important contributions was in the War Production Board during World War II, where he served as Head of the Specifications Branch of the Conservation Division, and later headed the Materials Division. This work resulted in tremendous savings of critical and strategic war materials through the issuance of emergency standards and in other ways.

He had written a large number of technical papers and reports dealing with properties and tests of engineering materials and especially standardization of specifications and tests.

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